Date: September 26, 2007

OU3 SOP 5 (Rev. 0)

Title: SEDIMENT SAMPLING

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		9/26/07
SOP Author		9/26/07

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1.0 INTRODUCTION

This Standard Operating Procedure (SOP) describes the protocols to be followed when sediment samples are collected for physical or chemical analysis. The procedures presented herein apply to sediment sampling from surface waters, wetlands, ponds, drainage structures, etc.

This document focuses on methods and equipment that are readily available and typically applied in collecting sediment samples. It is not intended to provide an all-inclusive discussion of sample collection methods. Specific sampling problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be clearly described in the project-specific sampling plan and approved by the Project Manager and the Quality Manager.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in sediment sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Environmental Sample: A solid sample collected for chemical or geotechnical analysis. These samples are used to support remedial investigation, feasibility studies, treatability studies, remediation design and performance assessment, waste characterization, etc.

Hand Auger: A sampling tool consisting of a stainless steel tube with two sharpened spiral wings at the tip.

Shovel/Trowel: A sampling device consisting of a stainless steel spade attached to a handle.

Core Sampler: A variable diameter stainless steel tube that can be attached to a hammer for

driving into sediment. The tube can also be fitted with retaining liners.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects site-specific sampling methods, sample locations, and constituents to be analyzed with input from other key project staff.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL, geologist, hydrogeologist, or engineer in the implementation of tasks. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

5.0 SEDIMENT SAMPLING PROCEDURES

This section describes sediment sampling from the bottom of a surface water drainage course or pond. The collected samples will be placed in appropriate sample containers, as designated by the FSP or Quality Assurance Project Plan (QAPP), for transfer to a laboratory for the analyses identified in the FSP. Details of sample collection will be described on the attached sediment sampling form.

5.1 Sampling Equipment and Methods

All sediments, including sediment submerged under water, may be sampled by the following methods:

A. Direct Sampling Method -- Fluid sediment may be collected directly using the sample container. If sampled under water, the container will be capped in place to avoid disturbance while surfacing.

B. Manual Core Sampler Method -- A slide-hammer core sampler with stainless steel liners may be used to recover a relatively undisturbed core sample of the sediment. An extension section may be added to reach sediment intervals in deeper waters.

C. Remote Scoop Method -- A sampling cup or container attached to a pole may be used to collect a sediment sample in deeper water or where a longer reach is needed.

D. Bottom Sampling Dredge Method -- A sampling dredge attached to a cable also may be used to recover sediment samples in deeper waters.

The selection of sampling equipment listed above depends on the site conditions and sample type required. In addition, the following equipment is needed to collect sediment samples:

- Field notebook, indelible marker
- Global Positioning System (GPS) unit
- Marking stakes
- Digital Camera
- Compass
- 100 m measuring tapes
- Detergent solution (0.1-0.3 % Alconox)
- Distilled water
- Latex gloves
- Ziploc bags
- Paper Towel
- Chain of custody and sample labels
- Coolers
- Sample bottles
- Plastic sheeting

5.1.1 Stream Sediment Sample Collection

Collect surface water samples according to SOP-3 prior to collecting sediment samples. Stream sediment samples will be composite samples comprised of five subsamples collected from the surface to a depth of 4 inches, which is the most biologically active sediment zone. The five subsamples will be collected from random locations along the creek channel within 200 feet of the specified location. No sediments will be collected from overbank areas, unless specified in the FSP.

For collecting sediment samples, the procedures outlined below shall be followed.

1. Don appropriate health and safety equipment.
2. Setup clean plastic sheeting in area for processing samples.
3. Collect sediment samples using decontaminated equipment from inundated areas beginning at the most downstream location (i.e., no sediments will be collected from overbank areas).
4. Collect approximately 200 grams of sediment from each subsampling location.
5. Composite the five subsamples in a water-tight plastic bag (e.g., Ziploc© bags) and homogenize by hand kneading.
6. Carefully remove twigs, rocks, leaves and other undesirable debris not considered part of the sample.
7. Carefully decant excess water from the sample bag, if necessary, taking care to avoid decanting fine sediment.
8. Double bag the sample. Label outer bag with the Index ID, sample location, and sample analysis information in accordance with the procedures in SOP No. 9. Place in cooler on ice for storage and shipment (refer to SOP-8 for sample handling and shipping information).

9. Complete the appropriate sediment Field Sample Data Sheet (FSDS) form to document the station and sample details (see SOP No. 9, Attachment 1). Document sediment characteristics, sample location as well as any changes to this SOP in the field logbook.

10. Locate the sample using a site map or GPS according to SOP-11.

For duplicates, a second sediment composite sample will be collected (Steps 3-9 above) from the sampling reach and placed into a new sample bag.

5.1.2 Storm Water Detention Pond Sediment Sample Collection

A single composite sediment sample will be collected at each of the storm water detention ponds designated in the FSP according to the steps presented in section 5.1.1. Sediment will be collected from the bottom surface, not from the side slopes, of the pond in areas that would most frequently contain storm water.

If water is present in the detention pond at the time of sampling, then procedures for collecting subaqueous sediment cores from a lake or a pond will be adopted. When cores are collected by wading, the sampler will proceed in a circular direction around the pond to collect the five subsamples and avoid disturbing sediments at the subsampling locations prior to use of the coring device. Core samples will be collected using a Wildco® K-B Corer or similar device with clear plastic liners. The core barrel will be advanced a minimum of 6 inches or to the depth of refusal, but only the shallowest 4 inches will be processed as a sample according to the steps presented in section 5.1.0. Once collected, each sediment core will be capped (with top and bottom marked).

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

Duplicate, equipment rinsate, and matrix spike samples will be collected at the frequencies documented in the field sampling plan. All sampling data must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and the other procedures referenced herein. All equipment must be

operated according to the manufacturer's specifications, including calibration and maintenance.

7.0 DECONTAMINATION

All equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

8.0 REFERENCES

Agency for Toxic Substances and Disease Registry (ATSDR). 2006. Asbestos Exposure and Your Health.

U.S. Environmental Protection Agency. 2007. Guidance for Preparing Standard Operating Procedures (SOPs). EPA QA/G-6. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/600/B-07/001. April 2007.

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007 OU3 SOP 7 (Rev. 0)

Title: EQUIPMENT DECONTAMINATION

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		9/26/07
SOP Author		9/26/07

Revision Number	Date	Reason for Revision
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1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is based on MWH SOP-02, Equipment Decontamination, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. Decontamination of drilling, sampling, and monitoring equipment is a necessary and critical aspect of environmental field investigations. Proper decontamination is a key element in reducing the potential for cross-contamination between samples from different locations, as well as ensuring that samples are representative of the sampled materials. Improper decontamination may result in costly re-collection and re-analysis of samples. All equipment used in the sampling process will be properly decontaminated prior to the collection of each sample and after completion of sampling activities.

The procedures outlined in this SOP will be followed during decontamination of field equipment used in the sampling process, including drilling, soil/water sample collection, and monitoring activities. Any deviations from these procedures will be noted in the field notebooks and approved by the appropriate oversight agency, if significant. Three major categories of field equipment, along with applicable decontamination methods for each, are discussed below.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in equipment decontamination must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Bailer: A cylindrical tool designed to remove material from a well. A valve at the bottom of the bailer retains the contents in the bailer.

Bladder Pump: Groundwater sampling equipment consisting of a flexible bladder, usually made of Teflon®, contained within a rigid cylindrical body (commonly made of stainless steel). The lower end of the bladder is connected to the intake port through a check valve, while the upper end is connected through a second check valve to a sampling line that leads to the ground surface.

Brass Sleeve: Hollow, cylindrical sleeves made of brass and used as liners in split-spoon samplers for collection of undisturbed samples.

Auger Flight: An individual auger section, usually 5 feet in length.

Continuous Core Barrel: 3-5 foot long steel barrels that can be joined together to allow continuous cores to be collected during a single run.

Drill Pipe: Hollow metal pipe used for drilling, through which soil and groundwater sampling devices can be advanced for sample collection.

Peristaltic Pump: A low-volume suction pump. The compression of a flexible tube by a rotor results in the development of suction.

Source Water: A drilling quality water source identified to be used for steam cleaning. This source should be sampled at the beginning of each field program to set baseline concentrations.

Distilled Water: Commercially available water that has been distilled. Each batch of distilled water should be analyzed to set baseline concentrations.

Hand Auger: A sampling tool consisting of a metal tube with two sharpened spiral wings at the tip.

Split-Spoon Sampler: A sampling tool consisting of a thick-walled steel tube with a removable head and drive shoe. The steel tube splits open lengthwise when the head and drive shoe are removed.

Scoop: A sampling hand tool consisting of a small shovel- or trowel-shaped blade.

Submersible Pump: Groundwater sampling pump that consists of a rotor contained within a chamber and driven by an electric motor.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Responsible for project implementation and coordination, selects project-specific drilling and sampling methods, and associated decontamination procedures with input from other key project staff, and appropriate oversight agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Geologist, Hydrogeologist, or Engineer: Implements the field program and supervises other sampling personnel, and ensures that SOPs are properly followed. Prepares daily logs of field activities.

Field Sampling Technician (or other designated personnel): Assists the FTL, geologist, hydrogeologist, or engineer in the implementation of tasks and is responsible for the decontamination of sampling equipment.

5.0 DECONTAMINATION PROCEDURES

Drilling and sampling procedures require that decontaminated tools be employed in order to prevent cross-contamination. The decontamination procedures described below will be followed to ensure that only uncontaminated materials will be introduced to the subsurface during drilling and sampling. The equipment decontamination process will be undertaken before and after each use of the equipment and include either steam cleaning or washing. Steam cleaning of equipment, if used, will be performed at a temporary decontamination site. The flooring of the temporary decontamination site will be impermeable to water and large enough to contain the equipment and the rinsate produced.

If the quantity of water in the pad area exceeds its holding capacity, the water will be drummed temporarily until analytical results are obtained and the water can be properly disposed of. Steam cleaning will not be performed over bare ground, but will always be conducted so that rinsate can be collected and disposed of properly. Wherever applicable, equipment will be disassembled to permit adequate cleaning of the internal portions.

5.1 Drilling and Large Equipment

The following procedure will be used for decontamination of large pieces of equipment. These include well casings, auger flights, drill pipes and rods, and those portions of the drill rig that may stand directly over a boring or well location, or that may come into contact with casing, auger flights, pipes, or rods.

- Establish a decontamination area large enough to contain the equipment and any decontamination waste
- Place equipment on sawhorse or equivalent, if possible.

- Steam clean the external surfaces and internal surfaces, as applicable, on equipment using high-pressure steam cleaner from an approved water source. If necessary, scrub using brushes and a phosphate-free detergent (e.g., Alconox™), or equivalent laboratory-grade detergent until all visible dirt, grime, grease, oil, loose paint, rust, etc., have been removed.

- Rinse with potable water

- Remove equipment from decontamination pad and allow to air dry

- Record date and time of equipment decontamination

5.2 Soil and Groundwater Sampling Equipment

The following procedure will be used to decontaminate sampling equipment such as split-spoon samplers; brass sleeves; continuous core barrels; scoops; hand augers; non-dedicated bailers; submersible pumps, bladder pumps; and other sampling equipment that may come into contact with samples. To minimize decontamination procedures in the field, dedicated equipment will be used wherever feasible:

- Wash and scrub equipment with phosphate-free, laboratory-grade detergent (e.g., Alconox™ or equivalent) and off-site distilled water

- Triple-rinse with distilled water

- Air dry

- Wrap in aluminum foil, or store in clean plastic bag or designated casing.

- Record date and time of equipment decontamination

Personnel involved in decontamination activities will wear appropriate protective clothing as defined in the project-specific health and safety plan.

5.3 Monitoring Equipment

The following procedure will be used to decontaminate monitoring devices such as slug-test equipment, groundwater elevation and free product thickness measuring devices, and water quality checking instruments. Note that organic solvents can not be used to decontaminate free product measuring devices because they will cause damage to the probes. Spray bottles may be used to store and dispense distilled water.

- Wash equipment with laboratory-grade, phosphate-free detergent (e.g., Alconox^{TM} or equivalent) and distilled water
- Triple-rinse with distilled water
- Store in clean plastic bag or storage case.
- Record date and time of equipment decontamination

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

All equipment decontamination must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and other procedures referenced herein.

To assess the adequacy of decontamination procedures, field rinsate blanks may be collected. The specific number of rinsate blanks will be defined in a FSP or work plan or by the Project

Manager. In general, at least one field rinsate blank should collected per sampling event or per day.

Rinsate blanks with elevated or detected contaminates will be evaluated by the Project Manager, who will relay the results to the site workers. Such results may be indicative of inadequate decontamination procedures that require corrective actions (e.g., retaining).

7.0 PROCEDURE FOR WASTE DISPOSAL

All decontamination water that has come into contact with contaminated equipment will be handled, labeled, stored and disposed according to SOP 12. Unless otherwise specified in the FSP, waste generated from other sources and classified as non-hazardous waste (e.g., PPE, pastic sheeting, rope and misc. debris) will be disposed into trash receptacles.

8.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. *Asbestos Exposure and Your Health*.

U.S. Environmental Protection Agency, *RCRA Ground-Water Monitoring: Draft Technical Guidance*, November 1992. Page 7-17.

Date: September 26, 2007

OU3 SOP 8 (Rev. 0)

Title: SAMPLE HANDLING AND SHIPPING

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		9/26/07
SOP Author		9/26/07

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1.0 INTRODUCTION

This standard operating procedure (SOP) is based on MWH SOP-09, Sample Handling and Shipping, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. This SOP describes the requirements for sample handling, storage and shipping. The purpose of this SOP is to define sample management activities as performed from the time of sample collection to the time they are received by the laboratory.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Chain-of-Custody: An accurate written record of the possession of each sample from the time of collection in the field to the time the sample is received by the designated analytical laboratory.

Sample: Physical evidence collected for environmental measuring and monitoring. For the purposes of this SOP, sample is restricted to solid, aqueous, air, or waste matrices. This SOP does not cover samples collected for lithologic description nor does it include remote sensing imagery or photographs (refer to SOP-9 for field documentation procedures).

Sampler: The individual who collects environmental samples during fieldwork.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, and etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: The Project Manager is responsible for ensuring that the requirements for sample management are included in the appropriate project plans. The Project Manager is responsible for coordinating sample management efforts with input from other key project staff and applicable government agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader and/or Field Hydrogeologist, Geologist or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Field Technician: Responsible for sample collection, documentation, packaging, and shipping. Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks.

5.0 PROCEDURES

5.1 Applicability

The information in this SOP may be used by direct reference or incorporated into project-specific plans. Deviations or modifications to procedures addressed herein must be brought to the attention of, and approved by, applicable government agencies.

5.2 Sample Management

Sample Containers: The sample containers to be used will be dependent on the sample matrix and analyses desired, and are specified in the project FSP. Only certified pre-cleaned sample containers will be used. Sample containers will be filled with adequate headspace (approximately 10 percent) for safe handling upon opening, except containers for volatile organic compound (VOC) analyses, which will be filled completely with no headspace. This no-headspace requirement applies to both soil and groundwater samples.

Once opened, the containers will be used immediately. If the container is used for any reason in the field (e.g., screening) and not sent to the laboratory for analysis, it will be discarded. Prior to discarding the contents of the used container and the container, disposal requirements will be evaluated. When storing before and after sampling, the containers will remain separate from solvents and other volatile organic materials. Sample containers with preservatives added by the laboratory will not be used if held for an extended period on the job site or exposed to extreme heat conditions. Containers will be kept in a cool, dry place. For preserved samples (except VOCs), the pH of the sample will be checked following collection of the sample. If the pH is not at the required level, additional preservative (provided by the laboratory) will be added to the sample container.

Numbering and Labeling: Refer to OU3 SOP-9.

Custody Seals. Custody seals with the date and initials of the sampler will be used on each shipping container to ensure custody. The custody seal will be placed on opposites sides of the cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of the liner bag inside of the cooler.

Chain-of-Custody: COC procedures require a written record of the possession of individual samples from the time of collection through laboratory analyses. A sample is considered to be in custody if it is:

- In a person's possession
- In view after being in physical possession
- In a secured condition after having been in physical custody
- In a designated secure area, restricted to authorized personnel

The COC record will be used to document the samples taken and the analyses requested. Refer to SOP-9 Attachment 2 for the OU3-specific COC form. Information recorded by field personnel on the COC record will include the following:

- Sample identifier (Index ID)
- Date and time of collection
- Sample matrix
- Preservation
- Type of analyses requested
- Unique COC number
- Lab being shipped to
- Signature of individuals involved in custody transfer (including date and time of transfer)
- Airbill number (if appropriate)
- Any comments regarding individual samples (e.g., organic vapor meter readings, special instructions).

COC records will be placed in a waterproof plastic bag (e.g., Ziploc®), taped to the inside lid of the cooler or placed at the top of the cooler, and transported with the samples. Signed airbills will serve as evidence of custody transfer between the field sampler and courier, as well as between the courier and laboratory. If a carrier service is used to ship the samples (e.g., Federal Express, etc.), custody will remain with the courier until it is relinquished to the laboratory. Upon receiving the sample cooler, a laboratory representative should sign in the receiving box of the COC, thus establishing custody. The sampler will retain copies of the COC record and airbill.

Sample Preservation/Storage: The requirements for sample preservation are dependent on the desired analyses and the sample matrix, and are specified in the FSP.

5.3 Sample Shipping

The methods and procedures described in this SOP were developed from these sources:

- 49 CFR 173. Shippers – Shippers – General Requirements for Shipping. United States Code of Federal Regulations available online at http://www.gpoaccess.gov/cfr/index.html
- 49 CFR 178. Specifications for Packaging. United States Code of Federal Regulations available online at http://www.gpoaccess.gov/cfr/index.html
- ASTM D 4220. Standard Practice for Preserving and Transporting Soil Samples. American Society for Testing and Materials available online at http://www.astm.org/
- ASTM D 4840. Standard Practice for Sampling Chain-of-Custody Procedures. American Society for Testing and Materials available online at http://www.astm.org/

Procedures for packaging and transporting samples to the laboratory are dependent on the chemical, physical, and hazard properties of the material. The procedures may also be based on an estimation of contaminant concentrations/properties in the samples to be shipped. Samples will be identified as environmental samples, excepted quantities samples, limited quantities samples, or standard hazardous materials. Environmental samples are defined as solid or liquid samples collected for chemical or geotechnical analysis. Excepted quantities involve the shipment of a few milliliters of either an acid or base preservative in an otherwise empty sample container. Limited quantities are restricted amounts of hazardous materials that may be shipped in generic, sturdy containers. Standard hazardous material shipments require the use of stamped/certified containers. All samples will be packaged and shipped or hand delivered to the laboratories the same day of sample collection, unless otherwise specified in the project-specific FSPs.

The following paragraphs describe standard shipping procedures for different types of samples. Any exceptions to these procedures will be defined in the FSP. It is the responsibility of the sampler to refer to the U.S. Department of Transportation (DOT) (http://hazmat.dot.gov/regs/rules.htm) regulations when dealing with a substance not addressed in this SOP for requirements and limitations associated with the shipment.

Sample Shipping via Commercial Carrier:

Aqueous or Solid Samples: Samples will be packaged and shipped to the laboratories the same day of sample collection, unless otherwise specified in the FSP and depending on holding time requirements for individual samples. For aqueous or solid samples that are shipped to the laboratory via a commercial carrier the following procedures apply:

- Sample labels will be completed and attached to sample containers.
- The samples will be placed upright in a waterproof metal (or equivalent strength plastic) ice chest or cooler.
- For shipments containing samples for volatile organic analysis, include a trip blank.
- Ice in double Ziploc® bags (to prevent leakage) will be placed around, among, and on top of the sample bottles. Enough ice will be used so that the samples will be chilled and maintained at 4°C ± 2°C during transport to the laboratory. Dry ice or blue ice will not be used.
- To prevent the sample containers from shifting inside the cooler, the remaining space in the cooler will be filled with inert cushioning material, such as shipping peanuts, additional bubble pack, or cardboard dividers, such that the sample containers remain upright and do not break.
- Tape shut the cooler's drain plug
- The original copy of the completed COC form will be placed in a waterproof plastic bag and taped to the inside of the cooler lid or placed at the top of the cooler.
- The lid will be secured by wrapping strapping tape completely around the cooler in two locations.

- Mark the cooler with arrow labels indicating the proper upright position of the cooler.

- Custody seals consisting of security tape with the date and initials of the sampler will be used on each shipping container to ensure custody. Two signed custody seals will be placed on the cooler, one on the front and one on the back.

- A copy of the COC record and the signed air bill will be retained for the project files.

- Affix a label containing the name and address of the shipper to the outside of the cooler

Hand-Delivered Samples: For aqueous or solid samples that will be hand carried to the laboratory, the same procedures apply.

Excepted Quantities: Usually, corrosive preservatives (e.g., hydrochloric acid, sulfuric acid, nitric acid, or sodium hydroxide) are added to otherwise empty sample bottles by the analytical laboratory prior to shipment to field sites. However, if there is an occasion whereby personnel are required to ship bottles with these undiluted acids or bases, the containers will be shipped in the following manner:

1. Each individual sample container will have not more than 30 milliliters of preservative.

2. Collectively, the preservative in these individual containers will not exceed a volume of 500 milliliters in the same outer box or package.
3. Despite the small quantities, only chemically compatible material may be placed in the same outer box, (e.g., sodium hydroxide, a base, must be packaged separately from the acids).

4. Federal Express will transport nitric acid only in concentrations of 40 percent or less.

5. A "Dangerous Goods in Excepted Quantities" label will be affixed to the outside of the outer box or container. Information required on the label includes:

   - Signature of Shipper
   - Title of Shipper
   - Date
   - Name and Address of Shipper
   - Check of Applicable Hazard Class
   - Listing of UN Numbers for Materials in Hazard Classes

Limited Quantities: Occasionally, it may become necessary to ship known hazardous materials, such as pure or floating product. DOT regulations permit the shipment of many hazardous materials in "sturdy" packages, such as an ice chest or cardboard box (not a specially constructed and certified container), provided the following conditions are met:

1. Each sample bottle is placed in a plastic bag, and the bag is sealed. Each VOC vial will be placed in a sealable bag. As much air as possible is squeezed from the bag before sealing. Bags may be sealed with evidence tape for additional security.

2. Or each bottle is placed in a separate paint can, the paint can is filled with vermiculite, and the lid is affixed to the can. The lid must be sealed with metal clips, filament, or evidence tape. If clips are used, the manufacturer typically recommends six clips.

3. The cans are placed upright in a cooler that has had the drain plug taped shut inside and outside, and the cooler is lined with a large plastic bag. Approximately 1 inch of adsorbent material sufficient to retain any liquid that may be spilled, is placed in the bottom of the liner. Only containers having chemically compatible material may be packaged in each cooler or other outer container.

4. The COC record is sealed inside a plastic bag and placed inside the cooler. The sampler retains one copy of the COC record. The laboratory will be notified if the

sample is suspected of containing any substance for which the laboratory personnel should take safety precautions.

5. The cooler is shut and sealed with strapping tape (filament type) around both ends. Two signed custody seals will be placed on the cooler, one on the front and one on the back. Additional seals may be used if the sampler and/or shipper consider more seals to be necessary. Wide, clear tape will be placed over the seals to ensure against accidental breakage.

6. The following markings are placed on the side of the cooler:

    - Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA Dangerous Goods Regulations [DGR])
    - UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
    - Shipper's name and address
    - Consignee's name and address
    - The words "LIMITED QUANTITY"
    - Hazard Labels (Column E, List of Dangerous Goods, Section 4, IATA DGR)
    - Two Orientation (Arrow) labels placed on opposite sides.

7. The Airbill/Declaration of Dangerous Goods form is completed as follows:

    - Shipper's name and address
    - Consignee's name and address
    - Services, Delivery & Special Handling Instructions
    - Cross out "Cargo Aircraft Only" in the Transport Details Box
    - Cross out "Radioactive" under Shipment Type
    - Nature and Quantity of Dangerous Goods

- Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA DGR)
- Class or Division (Column C, List of Dangerous Goods, Section 4, IATA DGR)
- UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
- Packing Group (Column F, List of Dangerous Goods, Section 4, IATA DGR)
- Subsidiary Risk, if any (Column D, List of Dangerous Goods, Section 4, IATA DGR)
- Quantity and type of packing (number and type of containers: for example, "3 plastic boxes", and the quantity per container, "2 L", is noted as "3 Plastic boxes X 2 L" This refers to 3 plastic boxes (coolers are referred to as plastic boxes) with 2 liters in each box.
- Packing Instructions (Column G, List of Dangerous Goods, Section 4, IATA DGR).
- Note: Only those Packing Instructions in Column G that begin with the letter "Y" may be used. These refer specifically to the Limited Quantity provisions.
- Authorization (Write in the words Limited Quantity)
- Emergency Telephone Number (List 800-535-5053. This is the number for INFOTRAC.)
- Printed Name and Title, Place and Date, Signature.

Standard Hazardous Materials: Shipment of standard hazardous materials presents the most difficulty and expense. However, there may be occasion whereby a hazardous material cannot be shipped under the Limited Quantity provisions, (e.g., where there is no Packing Instruction in Column G, List of Dangerous Goods, IATA Dangerous Goods Regulations, that is preceded by the letter "Y").

In such cases, the general instructions noted above but for non-Limited Quantity materials will apply, with one important difference: standard hazardous materials shipment requires the use of certified outer shipping containers. These containers have undergone rigid testing and are, therefore, designated by a "UN" stamp on the outside, usually along the bottom of a container's side. The UN stamp is also accompanied by codes specifying container type, packing group rating, gross mass, density, test pressure, year of manufacturer, state of manufacturer, and manufacturer code name. The transport of lithium batteries in Hermit Data Loggers is an example of a standard hazardous material where only a designated outer shipping container may be used.

5.4 Holding Times

The holding times for samples will depend on the analysis and the sample matrix. Refer to the FSP for holding times requirements.

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

All sample shipments must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that handling and shipment documentation has been completed per this procedure and other procedures referenced herein.

7.0 DECONTAMINATION

All shipment coolers shall be maintained clean of sampled material to avoid exposure during shipment. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

8.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Enforcement Considerations for Evaluations of Uncontrolled Hazardous Waste Disposal Sites by Contractors, Draft, Appendix D, April 1980.

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: May 29, 2008

OU3 SOP 9 (Rev. 3)

Title: FIELD DOCUMENTATION

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	/RPM	6/3/08
SOP Author		5/29/08

Revision Number	Date	Reason for Revision
0	09/26/2007	--
1	10/5/2007	• Add section for "Corrections and Modifications" and Field Modification Approval form (Attachment 3) • Update Labeling section and COC (Attachment 2) to reflect non-asbestos analysis and container details • Update FSDS forms (Attachment 1) based on field team input
2	02/22/2008	• Incorporate changes to FSDS forms (Attachment 1) based on field input • Remove OU3 phase specificity in SOP text
3	05/29/2008	• Incorporate changes to FSDS forms (Attachment 1) based on field input

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is a general guidance document for the required documentation to be completed by field personnel during field investigations. This SOP is based on MWH SOP-04, Field Documentation, Revision 1.0, March 2006, modified for use at the Libby Mine Site. Documentation in the form of field logbooks, reports, and forms shall be completed for every activity in the field. Records shall be maintained on a daily basis as the work progresses. All field documentation shall be accurate and legible because it is deliverable to the client as potentially a legal document.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the site health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006). All personnel engaged in soil sampling must follow health and safety protocols described in the health and safety plan.

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects project-specific field documentation with input from other key project staff.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Field Technician (or other designated personnel): Assists the FTL and/or field geologist, hydrogeologist, or engineer in the implementation of field tasks and field documentation.

Field Sample/Data Manager: Responsible for proper handling and shipping of all samples collected by the field crew, electronic data entry of field sample data sheet (FSDS) and chain-of-custody (COC) forms, and scanning/posting of field documentation PDFs (FSDS, COC, field logbooks, digital photographs) to a dedicated FTP site.

4.0 FIELD DOCUMENTATION PROCEDURES

Field documentation serves as the primary foundation for all field data collected that will be used to evaluate the project site. There are two main forms of field documentation – field logbooks and FSDS forms. All field documentation shall be accurate, legible and written in indelible black or blue ink. Absolutely no pencils or erasures shall be used. Incorrect entries in the FSDS forms or field logbooks will be corrected by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction.

4.1 Field Logbooks

The field logbook shall be a bound, weatherproof book with numbered pages, and shall serve primarily as a daily log of the activities carried out during the fieldwork. All entries shall be made in indelible black or blue ink. A field logbook shall be completed for each operation undertaken during the field tasks. To further assist in the organization of the field log books, the project name and the date shall be recorded on top of each page along with the significant

activity description (e.g., surface sample or soil boring number). All original field documentation shall be retained in the project files.

Skipped pages or blank sections at the end of a field log book page shall be crossed out with an "X" covering the entire page or blank section; "No Further Entries," initials, and date shall be written by the person crossing out the blank section or page. The responsible field team member shall write his/her signature, date, and time after the day's last entry.

Field activities vary from project to project; however, the concept and general information that shall be recorded are similar. The descriptions of field data documentation given below serve as an outline; individual activities may vary in documentation requirements. A detailed description of two basic example logbooks, suitable for documentation of field activities, is given below. These field logbooks include the FTL logbook and the field geologist/sampling team logbook.

FTL Logbook: The FTL's responsibilities include the general supervision, support, assistance, and coordination of the various field activities. As a result, a large portion of the FTL's day is spent rotating between operations in a supervisory mode. Records of the FTL's activities, as well as a summary of the field team(s) activities, shall be maintained in a logbook. The FTL's logbook shall be used to fill out daily/weekly reports and daily quality control reports (DQCRs), and therefore, shall contain all required information. Entries shall be preceded with time in military units for each observation. Items to be documented include:

- Record of tailgate meetings
- Personnel and subcontractors on job site and time spent on the site
- Field operations and personnel assigned to these activities
- Site visitors
- Log of FTL's activities: time spent supervising each operation and summary of daily operations as provided by field team members
- Problems encountered and related corrective actions
- Deviations from the sampling plan and reasons for the deviations
- Records of communications; discussions of job-related activities with the client, subcontractor, field team members, and project manager

- Information on addresses and contacts
- Record of invoices signed and other billing information
- Field observations

Field Geologist/Sampling Team Logbook: The field geologist or sampling team leader shall be responsible for recording the following information in a logbook:

- Health and Safety Activities
  - Calibration records for health and safety equipment (e.g., type of PID, calibration gas used, associated readings, noise dosimeters, etc.)
  - Personnel contamination prevention and decontamination procedures
  - Record of daily tailgate safety meetings
- Weather
- Calibration of field equipment
- Equipment decontamination procedures
- Personnel and subcontractors on job site and time spent on the site
- Station identifier
- Sampling activities
  - Sample location (sketch)
  - Equipment used
  - Names of samplers
  - Date and time of sample collection
  - Sample interval
  - Number of samples collected
  - Analyses to be performed on collected samples
- Disposal of contaminated wastes (e.g., PPE, paper towels, Visqueen, etc.)
- Field observations
- Problems encountered and corrective action taken
- Deviations from the sampling plan and reason for the deviations
- Site visitors

4.2 Field Sample Documentation

Sample Labels: A unique sample identification label shall be affixed to all sample containers. All samples will be labeled in a clear, precise way for proper identification in the field and for tracking in the laboratory. At the time of collection, each sample will be labeled with a unique 5-digit sequential identification (ID) number, referred to as the Index ID. The Index ID for all samples collected as part of OU3 sampling activities will have a two-character prefix specific to the sampling Phase (e.g., Phase 1 samples will have a "P1" prefix, P1-12345) as specified in the applicable SAP. Index ID labels will be ½ inch x 1 ¾ inch in size and pre-printed for use in the field. For each Index ID, multiple labels will be printed to allow for multiple containers of the same sample (i.e., for different analyses).

Index ID Label Example:

P1-12345

Each collection container will be labeled with a container label that enables the field team member to record the container specific details, such as the method of sample preparation (e.g., filtered/unfiltered), method of preservation, and the analytical methods that will be requested. Container labels will be 2 inch x 4 inch in size and pre-printed for use in the field. Any container-specific information shall be written in indelible ink.

Container Label Example:

Station ID: _____ Date/Time:_____
Index ID:

Media (circle one): AQ SO AA BK DB TC
For AQ, Filtered? (circle one): Yes No
Container: _____
Preservation: _____
Analyses: _____
_____
Lab QC: _____ __ ☐ Archive

Media acronyms: AQ – aqueous media, SO – solid media, AA – ambient air, BK – tree bark, DB – organic debris, TC – tree age core

After labels have been affixed to the sample container, the labels will be covered with clear packaging tape to ensure permanence during shipping.

Any unused Index ID labels should be crossed out to avoid the possibility of using unused labels for a different sample.

Field Sample Data Sheet (FSDS) Forms: Data regarding each sample collected as part of the OU3 sampling will be documented using Libby-specific FSDS forms (provided as Attachment 1). These FSDS forms are media-specific and designed to facilitate data entry of station location, sample details, and field measurements needed for the OU3 investigation.

In the field, one field team member will be responsible for recording all sample details onto the appropriate FSDS form. At the time of sample labeling, one Index ID label will be affixed to the FSDS form in the appropriate field. All written entries on the FSDS form shall be accurate, legible and written in indelible black or blue ink.

Once the FSDS form is complete, written entries will be checked by a second field team member. These two field team members will initial the bottom of the FSDS form in the appropriate field to document who performed the written data entry and who performed the QC check of the FSDS form.

On a weekly basis (or more frequently as conditions permit), information from the hard copy FSDS form will be manually entered into a field-specific OU3 database using electronic data entry screens by the Field Sample/Data Manager. Once electronic data entry is complete, QC of all data entry will be completed by the FTL or their designate. The Field Sample/Data Manager and the FTL will initial in the appropriate field on the paper FSDS form to document who performed the data entry into the database and who performed the QC check.

4.3 Photologs

Photologs are often used in the field to document site conditions and sample location characteristics. While photographs may not always be required, they shall be used wherever applicable to show existing site conditions at a particular time and stage of the investigation or related site activity. Photolog information shall include:

- station location identifier
- Index ID (if applicable)
- date and time of photo
- direction/orientation of the photo
- description of what the photo is intended to show

An engineer's scale or tape shall be included in any photographs where scale is necessary. Any wasted frames or images in a roll of film or sequence of digital images shall be so noted in the field logbook.

4.4 Chain-of-Custody Records

Custody Seals: Custody seals with the date and initials of the sampler will be used on each shipping container to ensure custody. The custody seal will be placed on opposites sides of the cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of the liner bag inside of the cooler.

Chain-of-Custody Forms: COC procedures allow for the tracking of possession and handling of individual samples from the time of field collection through to laboratory analysis. Documentation of custody is accomplished through a COC form that lists each sample and the individuals responsible for sample collection and shipment, sample preparation, and receipt by the analytical laboratory. The COC form also documents the analyses requested for each sample. Whenever a change of custody takes place, both parties will sign and date the COC form, with the relinquishing party retaining a copy of the form. The party that accepts custody will inspect the COC form and all accompanying documentation to ensure that the information is complete and accurate. Any discrepancies will be noted on the COC form. Shipping receipts shall be signed and filed as evidence of custody transfer between field sampler(s), courier, and laboratory.

Attachment 2 provides an example of the COC form that will be used for all samples collected as part of OU3 sampling. This form will be printed as a carbonless triplicate form to facilitate

retention of COC copies by relinquishing parties. As seen, the COC form includes the following information:

- sample identifier (Index ID)
- date and time of collection
- method of sample preparation and preservation
- number of sample containers
- analyses requested
- shipping arrangements and airbill number, as applicable
- recipient laboratories
- signatures of parties relinquishing and receiving the sample

On a daily basis, the Field Sample/Data Manager will package samples for shipping, complete hard copy COC forms, and ship all samples as outlined in SOP No. 8. On a daily basis, information from the hard copy COC form necessary for sample tracking will be manually entered into a field-specific OU3 database using electronic data entry screens by the Field Sample/Data Manager. Once electronic data entry is complete, QC of all data entry will be completed by the FTL or their designate.

5.0 FIELD DATA TRANSMITTAL

Copies of all FSDS forms, COC forms, and field log books will be scanned and posted in portable document format (PDF) to a project-specific file transfer protocol (FTP) site daily. This FTP site will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related personnel. File names for scanned FSDS forms, COC forms, and field log books will include the sample date in the format YYYYMMDD to facilitate document organization (e.g., FSDS_20070831.pdf).

Electronic copies of all digital photographs will also be posted weekly (or more frequently as conditions permit) to the project-specific FTP site. File names for digital photographs will

include the station identifier, the sample date, and photograph identifier (e.g., ST-1_20070831_12459.tif).

A copy of the field-specific OU3 database will be posted to the project-specific FTP site on a weekly basis (or more frequently as conditions permit). The field-specific OU3 database posted to the FTP site will include the post date in the file name (e.g., FieldOU3DB_20070831.mdb).

6.0 CORRECTIONS AND MODIFICATIONS

6.1 Field Deviations and Modifications

It is recognized that deviations and modifications from the standard operating procedures may be necessary based on site conditions. Any requested field modifications will be submitted by Robert Marriam (Remedium Group, Inc. - W.R. Grace contractor) to Bonita Lavelle (EPA Region 8 - Remedial Project Manager) for review and approval. All modification requests will be recorded in a Field Modification Approval Form (see Attachment 3).

6.2 Corrections to Hard Copy Forms

If an error is identified on an FSDS or COC form prior to entry into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. Data entry into the field-specific OU3 database and scanning/posting of the hard copy forms should proceed following the data entry procedures described above.

If an error is identified on an FSDS or COC form after entry into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. The corrected form should be scanned and posted to the project-specific FTP site. File names for corrected FSDS forms will include the Index ID of the corrected sample to facilitate document organization (e.g., FSDS_C_P1-12345.pdf). File names for corrected COC forms will include the COC ID of the corrected COC form to facilitate document organization

(e.g., COC_C_OU3-36512.pdf). Necessary data corrections will be made to the master OU3 database by the database manager.

If changes are made to a COC form, the analytical laboratory should be provided with a corrected COC form.

7.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

RCRA Ground-Water Monitoring: Draft Technical Guidance, November 1992.

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ATTACHMENT 1

OU3 FIELD SAMPLE DATA SHEET (FSDS) FORMS

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FSDS Rev. 3

Sheet No.: SWS2- ____________
SED2- ____________

LIBBY OU3 PHASE II FIELD SAMPLE DATA SHEET
SURFACE WATER AND SEDIMENT

Station ID: ____________________ **Sampling Date**: ______________

Field Logbook ID: ______________________________________ **Logbook Page No**: ___________

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

For New Stations Only: **X coord:** ____________ **Y coord:** ____________ **Elev:** ____________

Sampling Team: **MWH** **Samplers Initials:** ____

WATER QUALITY PARAMETERS (if applicable)

Time Measured (hh:mm)	Temp. (°C)	pH	Specific Conductance (mS/cm Auto-comp @ 25°C)	Diss. O_2 (mg/L)	ORP (mV)	Turbidity (NTU)

SAMPLE COLLECTION

Index ID	*AFFIX LABEL HERE*	**Sampling Time:**__________________ **Sample Type: Field Sample** **Media : Surface Water Sediment**	**Sampling Method** (if applicable)**:** **Grab or Composite** **# of Composites:**___________ **Sampling Depth: Top (in)**_________ **Bot (in)**_________
Index ID	*AFFIX LABEL HERE*	**Sampling Time:**__________________ **Sample Type: SP FD** **MS MSD PE** **FB TB EB** **Media : Surface Water Sediment**	**Sampling Method (if applicable):** **Grab or Composite** **# of Composites:**___________ **Sampling Depth: Top (in)**_________ **Bot (in)**_________
Index ID	*AFFIX LABEL HERE*	**Sampling Time:**__________________ **Sample Type: SP FD** **MS MSD PE** **FB TB EB** **Media : Surface Water Sediment**	**Sampling Method (if applicable):** **Grab or Composite** **# of Composites:**___________ **Sampling Depth: Top (in)**_________ **Bot (in)**_________
Index ID	*AFFIX LABEL HERE*	**Sampling Time:**__________________ **Sample Type: SP FD** **MS MSD PE** **FB TB EB** **Media : Surface Water Sediment**	**Sampling Method (if applicable):** **Grab or Composite** **# of Composites:**___________ **Sampling Depth: Top (in)**_________ **Bot (in)**_________

COMMENTS

Note:						
	FS	Field Sample	**SP**	Field Split Sample	**FD**	Field Duplicate Sample
	TB	Trip Blank Sample	**MS**	Matrix Spike Sample	**MSD**	Matrix Spike Duplicate Sample
	FB	Field Blank Sample	**EB**	Equipment Decon Blank Sample	**PE**	Performance Evaluation Sample

Field Data Entered by:		**Field Entries Checked by:**	

Database Entry:		*Database QC:*

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ATTACHMENT 2

OU3 CHAIN OF CUSTODY FORM

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LIBBY OU3 – PHASE II CHAIN-OF-CUSTODY RECORD/REQUEST FOR ANALYSIS

COC No. ________________

PAGE: _______ OF: _______

ENTERED BY (Signature): ______________________________ PROJECT MANAGER: __________________________________ DATE: ____________________

METHOD OF SHIPMENT: ______________________________ CARRIER/WAYBILL NO.: ________________DESTINATION: ______________________________

SAMPLES							ANALYSIS REQUEST																													
							Asbestos			Non-Asbestos (a)																										
Index ID	Date	Time	Media*	Air Volume (L) or Tree Bark Sample Area (cm^2)	Filtered	Archive	TEM-ISO 10312 (b,c)	PLM (d)		TAL Metals+Boron	Mercury	TOC	DOC	Paste pH	Fluoride	Chloride, Sulfate	Total Phosphorus	Cyanide	VPH	EPH	OPP Pesticides	Chlorinated Pesticides	Herbicides	PCBs	SVOCs	VOCs	TDS, TSS, Nitrite, Alkalinity	Ammonia, Nitrate, TKN	Orthophosphate	Radiochemistry	Radium, Uranium	Hardness			Age core (e)	Remarks

	TOTAL NUMBER OF CONTAINERS	LABORATORY COMMENTS/CONDITION OF SAMPLES	Cooler Temp:

RELINQUISHED BY:			DATE	TIME	RECEIVED BY:		
SIGNATURE	PRINTED NAME	COMPANY			SIGNATURE	PRINTED NAME	COMPANY

** Media: AQ - Aqueous SO – Solid AA – Ambient Air BK – Tree Bark DB – Organic Debris (Duff) TC – Tree Age Core*

Notes --

(a) Method, container, and preservation details are provided in the attached tables

(b) With Libby-specific modifications. See applicable O3 SAP for counting and stopping rules

(c) For tree bark, preparation by TREE-LIBBY-OU3. For organic debris (duff), preparation by DUFF-LIBBY-OU3.

(d) Preparation by ISSI-LIBBY-01 and analysis by SRC-LIBBY-01 (PLM-Grav) and SRC-LIBBY-03 (PLM-VE)

(e) In accordance with procedures in Phipps (1985).

DISTRIBUTION: PINK: Field Copy YELLOW: Laboratory Copy WHITE: Return to Originator

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ATTACHMENT 3

OU3 FIELD MODIFICATION APPROVAL FORM

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FIELD MODIFICATION APPROVAL FORM
LFM-OU3-_____

Libby OU3 Phase II Sampling & Analysis Plan

Requested by:_____ Date:_____

Description of Deviation:

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

☐ EPA Region 8 has reviewed this field modification approves as proposed.

☐ EPA Region 8 has reviewed this field modification and approves with the following exceptions:

_____
_____
_____
_____
_____
_____
_____

☐ EPA Region 8 has reviewed this field modification and does not agree with the proposed approach for the following reasons:

_____
_____
_____
_____
_____
_____

_____ _____

Bonita Lavelle, EPA RPM Date

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Date: January 31, 2008

OU3 SOP 10 (Rev 1)

Title: FIELD EQUIPMENT CALIBRATION

APPROVALS:

EPA Remedial Project Manager — Date: 3/20/08

SOP Author — Date: 3/20/2008

Revision Number	Date	Reason for Revision
0	9/26/07	
1	1/31/08	Update based on field team input

LIBBY ASBESTOS SUPERFUND SITE OU3

STANDARD OPERATING PROCEDURE No.10

FIELD EQUIPMENT CALIBRATION

1.0 INTRODUCTION

This standard operating procedure (SOP) provides general guidelines on calibration and operating procedures for typical field equipment. This SOP is based on two documents: (1) MWH FMC SOP-01, Field Equipment Calibration, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3 and (2) USGS Survey Techniques of Water-Resources Investigations, Book 9. Field monitoring instruments are used to measure chemical parameters in situ and when data quality objectives specify screening-level analytical support. Screening-level data are collected for on-site, real-time measurements; evaluation of existing conditions; refinement of sampling locations; and health and safety evaluations. Field measurements are generally used to refine sampling programs and to estimate the extent of contamination at the site. This type of support also provides real-time data for health and safety purposes.

The purpose of this SOP is to define the calibration and operating procedures for equipment used for field monitoring.

2.0 DEFINITIONS

Conductivity: Is a measure of the quantity of electricity transferred across a unit area, per unit potential gradient, per unit time. Conductivity is measured by dipping a probe directly into the water source or into a separate sample aliquot.

Dissolved Oxygen (DO): Is a measure of the quantity of oxygen dissolved in water. DO data is collected in the field using direct measurement probes.

pH: Is measured directly using a probe and is the acidity or alkalinity of a solution; numerically equal to 7 for neutral solutions, increasing with increasing alkalinity and decreasing with increasing acidity.

Temperature: An indicator of the thermal energy contained in a solid or fluid. Units are degrees Centigrade (°C) or degrees Fahrenheit (°F). Temperature measurements are made with a mercury-filled thermometer, bimetallic-element thermometer, or electrical thermistor.

Turbidity: a measure of cloudiness in water due to suspended and colloidal organic and inorganic material. Turbidity is measured by using a field portable nephlometer capable of reading down to 0.1 Nephelometric Turbidity Units (NTU).

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Responsible for identifying the appropriate equipment necessary for adequate site characterization and the requirements for the project-specific tasks.

Quality Control Manager: Performs field program audits and ensures project data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the field program, and supervises other field staff to ensure proper calibration and use of field equipment through the duration of the project.

Field Technician (or other designated personnel): Responsible for providing requested instrumentation and basic instructions on its calibration and use. Assists the FTL with the implementation of tasks and is responsible for regular equipment maintenance and calibration.

4.0 GUIDELINES

This SOP provides a summary of the calibration and operating procedures in accordance with the various manufacturers' instruction manuals, which accompany each piece of equipment. This SOP will be reviewed and used in conjunction with the manufacturer's instruction manual by field team members when using field equipment.

4.1 APPLICABILITY

Field equipment must be kept in designated cases, packaged properly, and secured during transport to prevent equipment damage, which may result in inaccurate readings. Decontaminate and calibrate all equipment prior to use. As part of the calibration process, standard laboratory procedures of decontamination shall be followed; prior to calibration and between calibration buffers, solution vessels and probes shall be rinsed a minimum of three times with distilled/deionized (DI) water and a minimum of one time with the calibration buffer solution or sample solution.

Always calibrate meters according to the manufacturer's instructions before the start of each workday and whenever equipment drift is suspected. Consult the specific instruments' instruction manual for further calibration details.

4.2 pH METERS

Determining pH is critical for predicting and interpreting the reactions and migration of dissolved chemical constituents in groundwater or surface water. Whenever groundwater or surface water samples are collected, pH may be measured using a flow-

through type meter or another type of pH probe. Meters used will have temperature and slope adjustments and a repeatability of ± 0.01 standard pH units.

4.2.1 Calibration: Calibration standard buffers shall not be used beyond the date specified by the manufacturer, and calibration standard buffers shall be stored in a manner that protects the integrity and precision of the solution. Proper decontamination of equipment shall be performed following standard lab practices (refer to section 4.1) prior to calibration.

Two pH standard buffers will be used for calibration of the electrode and are to bracket the anticipated pH of the water samples. For example, if the anticipated pH of sample water is 6, calibration will be conducted with pH 4 and pH 7 buffer solutions; for an anticipated pH of 8, calibrate with pH 7 and pH 10 buffers. Three buffer solutions can be used to calibrate over a larger pH range. Because pH is temperature dependent, buffers and samples should be kept at similar temperature. The temperature of buffer solutions must be known, and temperature-correction factors must be applied before calibration adjustments are made. Theoretically, buffer solutions are stable indefinitely; however, they are susceptible to contamination. Therefore, old, partially full bottles will be replaced and solutions will not be used past the manufacturer's recommended expiration date. The instrument calibration will be checked periodically against a standard solution.

Meters with microprocessors have reliable autocalibration functions and will automatically compensate for buffer temperatures and indicate Nernst slope. For such meters, follow the manufacturer's calibration instructions precisely and completely.

- Check the records of electrode performance before each calibration and sampling event. Electrode response is optimum between approximately 98 percent and 99.5 percent. A slope of 94 percent indicates possible electrode deterioration. Do not use the electrode is the response slope is below 90 percent.
- Calibrate or check the temperature sensor calibration at least annually, and tag the sensor with the date of in-house certification. Do not use the automatic

temperature compensating function of a pH meter if it has not been certified within the past 12 months.

- Record calibration in the instrument log book and on field forms at the time of instrument calibration.

Procedure: Calibration and operating procedures differ with instrument systems—always check the manufacturer's instructions.

1. Equilibrate Equipment to Temperature (this is recommended, even if using an automatic compensating meter).

a. Bring the pH buffers, thermometer (if necessary), container, and electrode to the temperature of the sample.

- To equilibrate to stream temperature, place the buffer bottles in a minnow bucket or mesh bag and suspend them in the stream.

b. Allow 15 to 30 minutes for the buffers to adjust to the sample temperature.

- When making temperature corrections, use the correction factors provided by the buffer manufacturer (temperature coefficients can vary with buffer manufacturer).

2. Inspect the pH Electrode.

a. Check for damage to the electrode bulb, body, or cables.

b. Rinse any precipitate off of the electrode with DI water (the measurement can be affected if precipitate falls into the buffer or sample).

c. Slide the protective sleeve up or down to uncover the filling hole.

d. Gently shake or tap the electrode to dislodge and remove air bubbles trapped in the sensing tip of the electrode and to remove excess deionized water. Do not wipe the electrode.

3. Calibration Rinse.

a. Rinse the electrode, thermometer or automatic temperature compensating (ATC) sensor, and a container large enough to hold the sensors and buffer with pH buffer 7. Discard the used pH buffer into a waste container.

b. Pour fresh pH 7 buffer into the buffer-rinsed container that holds the electrode and thermometer. Allow the instruments to equilibrate for 1 minute (if necessary), then discard the buffer into a waste container.

4. Calibration.

a. Pour fresh pH 7 buffer into the container that holds the electrode and thermometer or ATC sensor.

- The bulb of the pH electrode must not touch the bottom or side of the container.
- Add enough pH buffer to cover the reference junction.

b. Swirl the sample gently or stir carefully with the electrode. If using a magnetic stirrer, stir slowly enough so that a vortex is not created. Place a thin piece of insulating material (styrofoam or cardboard) between the magnetic stirrer and beaker to prevent transfer of heat to the buffer solution.

c. Measure the temperature of the buffer solution; remove the thermometer (it is not necessary to remove the ATC sensor).

d. Determine the theoretical pH of the buffer from the temperature-correction tables.

e. Note and record the pH temperature readings. Adjust the meter reading to the pH value using the "standardize" function on the meter (usually a knob or pressure pad). Record the adjusted pH value for the 7.0 buffer and associated millivolt reading.

f. Remove the electrode and ATC sensor (some instruments require that the meter be switched to the standby or off position before removing the electrode from the solution).

- Repeat the calibration steps using fresh portions of reference buffer solution until two successive readings are obtained at the adjusted pH value for pH 7 buffer without further adjustment to the system.

- Discard the used pH 7 buffer into a waste container.

5. Slope Adjustment Rinse.

a. Rinse the electrode and thermometer or ATC sensor thoroughly with DI water.
b. Rinse a clean container, electrode, and thermometer with a second buffer (usually pH 4 or 10) that brackets the expected pH value of the sample; discard the used buffer into a waste container.
c. Pour the second buffer into a container holding the electrode and thermometer or ATC sensor. Allow the temperature to equilibrate for 1 minute, then discard the used buffer into a waste container.

6. Slope Adjustment. ***Note: For most modern meters, this step is automated—if so, skip to step seven.

a. Pour a fresh portion of the second pH buffer into a container holding the electrode and thermometer or ATC sensor.
b. Stir slowly (no vortex) or swirl manually. Follow the directions in 4b, above.

c. Measure the temperature and pH of the buffer solution and check the pH value of the buffer on temperature coefficient tables. Record the pH and temperature readings.
d. Adjust the slope to the value of the second pH buffer at known temperature. (Some meters have separate slope-adjustment knobs, pressure pads, or other devices, whereas others have to be adjusted by use of a temperature knob.) Record the adjusted pH value and associated millivolt reading.
e. Discard the used buffer into a waste container.
f. Repeat steps 6(a) through 6(e) using successive portions of the buffer solution until two successive readings are obtained without further adjustment.

7. Rinse the electrode and thermometer or ATC sensor thoroughly with DI water.

8. If using a non-compensating or non-automated meter, repeat the calibration rinse (step 3) and calibration procedures [steps 4(a) through 4(d)] to ensure that the slope adjustments did not affect the calibration adjustment.

- This step is a check only; no adjustment should be needed, but the result should be recorded. If adjustment is needed, repeat the entire calibration procedure.
- If adjustment is still needed, a systematic problem is likely (see 6.4.4). Inspect the instrument system, clean the electrode or add filling solution, or use a spare electrode or meter.

9. Calibration Check Rinse.

a. Rinse the electrode and thermometer or ATC sensor with DI water.

b. Rinse another clean container, electrode, and thermometer with a third buffer (pH 4 or 10) and discard the used buffer into a waste container.

c. Pour the third buffer into a container holding the electrode and thermometer or ATC sensor. Allow the temperature to equilibrate for 1 minute, then discard the used buffer into a waste container.

10. Calibration Range Check.

a. Pour a fresh portion of third pH buffer into a container holding the electrode and thermometer or ATC sensor.

b. Stir without forming a vortex or swirl slowly (see step 4b).

c. Measure the temperature of the buffer solution (remove the liquid-filled thermometer and check the temperature-adjusted pH value), if necessary for the meter being used.

d. The pH instrument system should read the value of the third buffer at a known temperature within ±0.1 pH units.

- Meters reading to three or more places to the right of the decimal may not provide better accuracy than ±0.05 units, and their accuracy must be verified.

- If it checks, the instrument system is calibrated over a range of pH 4 to 10 and is ready for ANC or alkalinity titrations as well as pH measurement.
- If the instrument system does not check over the entire range, recalibrate before measuring the sample pH. Recalibrate before an alkalinity/ANC titration if the sample has a pH greater than 7.0.

e. Discard the used buffer into a waste container.

f. Rinse the electrode and thermometer (or ATC sensor) with DI water

4.2.2 Measurement

It is generally preferable to measure pH in situ rather than on a sample taken from a splitter or compositing device. If stream conditions are such that water would pass the in situ pH sensor at a very high rate of flow, however, streaming-potential effects could affect the accuracy of the measurement. For such conditions, it is preferable to withdraw a discrete sample directly from the stream or compositing device and use the sub-sample measurement procedures described below. The pH instrument system should be set up on board the boat or on-shore so that pH is measured at the time of sample collection.

The pH of a water sample can change significantly within hours or even minutes after sample collection as a result of degassing (such as loss of carbon dioxide, hydrogen sulfide, and ammonia); mineral precipitation (such as formation of calcium carbonate); temperature change; and other chemical, physical, and biological reactions. The electrometric method of pH measurement described below applies to filtered or unfiltered surface water and ground water, from fresh to saline.

Field conditions, including rain, wind, cold, dust, and direct sunlight can cause measurement problems. To the extent possible, shield the instrument and measurement process from the effects of harsh weather.

Record the pH variation from a cross-sectional profile, if possible, to determine if pH is uniform at any given discharge, and select the sampling method appropriate for study objectives.

- To sample a shallow flowing stream, wade to the location where pH is to be measured.
- To sample a stream or river too deep to wade, lower a weighted pH sensor with a calibrated temperature sensor (if needed) from a bridge, cableway, or boat. Do not attach the weight to a sensor or sensor cables.
- To sample under still water conditions, measure pH at multiple depths at several points in the cross-section.

Procedure:

1. Ensure that the instrument is calibrated.
2. Immerse the pH probe in the water to the correct depth and hold it there for at least 60 seconds to allow for temperature equilibration.
3. Record the pH and temperature values without removing the sensor from the water.
    - Values generally stabilize quickly within ±0.05 to 0.1 standard pH unit, depending on the instrument system.
    - Record the median of the observed values.
    - If readings do not stabilize after extending the measurement period, note this on the field forms along with the pH readings, and record the median value of the last five or more readings.
    - After measurements are completed, rinse the pH probe with deionized water and continue with further sampling.
4. For EWI (Equal Width Increment) or EDI (Equal Depth Increment) measurements—Proceed to the next station in the cross section. Repeat steps 3 through 5. After all stations in the cross section have been measured, rinse the sensors with deionized water and store them.
5. Record the mean or median stream pH on the field forms
    - **In still water**—median of three or more sequential values.

- **EDI**—mean value of all subsections measured (use the median if measuring one vertical at the centroid of flow).
- **EWI**—mean or median of all subsections measured.

4.3 SPECIFIC CONDUCTANCE METERS

Specific conductance is used as an indicator of water quality. It is a simple indicator of the change within a system and provides useful information for site characterization. Any meter used to collect field specific conductance measurements will be equipped with a temperature compensator, and read directly in micromhos per centimeter (µmhos/cm) corrected to 25°C. The meter will be calibrated to record values over the anticipated range of conductivity values during measurement.

4.3.1 Calibration

Reagent-grade potassium chloride (KCl) will be used for the calibration of specific conductance equipment. Calibration standards will not be used beyond the date specified by the manufacturer. Consult the manufacturer's instruction manual for further details. Specific conductance readings will be reported on the field logs in micromhos/centimeter (µm/cm) or millimhos/cm. The instrument calibration will be checked before every water-quality field trip and periodically throughout the sampling event against a standard solution of KCl.

Procedure: Calibration and operating procedures differ with instrument systems—always check the manufacturer's instructions.

1. Inspect the instrument and the conductivity sensor for damage. Check the battery voltage. Make sure that all cables are clean and connected properly.
2. Turn the instrument on and allow sufficient time for electronic stabilization.
3. Select the correct instrument calibration scale for expected conductivity.

4. Select the sensor type and the cell constant that will most accurately measure expected conductivity.

5. Select two conductivity standards that will bracket the expected sample conductivity. Verify that the date on the standards has not expired.

6. Equilibrate the standards and the conductivity sensor to the temperature of the sample.

   a. Put bottles of standards in a minnow bucket, cooler, or large water bath that is being filled with ambient water.

   b. Allow 15 to 30 minutes for thermal equilibration. Do not allow water to dilute the standard.

7. Rinse the conductivity sensor, the thermometer (liquid-in-glass or thermistor), and a container large enough to hold the dip-type sensor and the thermometer.

   a. First, rinse the sensor, the thermometer, and the container three times with deionized water.

   b. Next, rinse the sensor, the thermometer, and the container three times with the standard to be used.

8. Put the sensor and the thermometer into the rinsed container and pour in fresh calibration standard.

9. Measure water temperature. Accurate conductivity measurements depend on accurate temperature measurements or accurate temperature compensation.

   a. If the sensor contains a calibrated thermistor, use this thermistor to measure water temperature.

   b. If using a manual instrument without a temperature display or temperature compensation, adjust the instrument to the temperature of the standard using a calibrated liquid-in-glass or a thermistor thermometer.

10. Agitate a submersible-type conductivity sensor up and down under the solution surface to expel air trapped in the sensor. Read the instrument display. Agitate the sensor up and down under the solution surface again, and read the display. Repeat the procedure until consecutive readings are the same.

11. Record the instrument reading and adjust the instrument to the known standard value.

    a. For non temperature-compensating conductivity instruments, apply a temperature-correction factor to convert the instrument reading to conductivity at 25°C.

    b. The correction factor depends to some degree on the specific instrument used—use the temperature-correction factor recommended by the manufacturer.

    c. If an instrument cannot be adjusted to a known calibration standard value, develop a calibration curve. After temperature compensation, if the percentage difference from the standard exceeds 5 percent, refer to the instrument troubleshooting guide.

12. Record in the instrument log book and on field forms:

    a. The temperature of the standard solution.

    b. The known and the measured conductivity of the standard solution (including ± variation).

    c. The temperature-correction factor (if necessary).

13. Discard the used standard into a waste container. Thoroughly rinse the sensor, thermometer, and container with deionized water.

14. Repeat steps 7 through 13 with the second conductivity standard.

    a. The purpose for measuring a second standard is to check instrument calibration over the range of the two standards.

    b. The difference from the standard value should not exceed 5 percent.

    c. If the difference is greater than 5 percent, repeat the entire calibration procedure. If the second reading still does not come within 5 percent of standard value, refer to a troubleshooting guide or calibrate a backup instrument.

    d. Switching instrument calibration scales could require re-calibration.

15. Record in the instrument log book and on field forms the calibration data for the second standard.

4.3.2 Measurement

In situ measurement is preferred for determining conductivity of surface water. Conductivity measurements should be performed immediately upon sample collection at the field site.

Field conditions (rain, wind, cold, dust, direct sunlight) can cause measurement problems—Shield the instrument to the extent possible and perform measurements in a collection chamber in an enclosed vehicle or an on-site laboratory.

For waters susceptible to significant gain and loss of dissolved gases, make the measurement within a gas-impermeable container (Berzelius flask) fitted with a stopper—Place the sensor through the stopper and work quickly to maintain the sample at ambient surface-water or ground-water temperature.

Avoid contamination from the pH electrode filling solution—Measure conductivity on a separate discrete sample from the one used for measuring pH; in a flowthrough chamber, position the conductivity sensor upstream of the pH electrode.

The conductivity measurement reported must account for sample temperature. If using an instrument that does not automatically temperature compensate to 25°C, record the uncompensated measurement in your field notes, along with the corrected conductivity value. Use correction factors supplied by the instrument manufacturer.

Conductivity measurements in flowing surface water should represent the cross-sectional mean or median conductivity at the time of observation. Any deviation from this convention must be documented in the data base and with the published data.

Before beginning, take a cross-sectional conductivity profile to determine the degree of system variability if feasible. A submersible sensor works best for this purpose.

Procedure:

1. Calibrate the conductivity instrument system at the field site after equilibrating the buffers with stream temperature.

2. Record the conductivity variation from a cross-sectional profile on a field form and select the sampling method.

- Flowing, shallow stream—wade to the location(s) where conductivity is to be measured.
- Stream too deep or swift to wade—lower a weighted conductivity sensor from a bridge, cableway, or boat. Do not attach weight to the sensor or the sensor cable.
- Still-water conditions—measure conductivity at multiple depths at several points in the cross section.

3. Immerse the conductivity and temperature sensors in the water to the correct depth and hold there (no less than 60 seconds) until the sensors equilibrate to water conditions.

4. Record the conductivity and corresponding temperature readings without removing the sensors from the water.

- Values should stabilize quickly to within 5 percent at conductivity ≤100 µS/cm and within 3 percent at conductivity >100 µS/cm.
- Record the median of the stabilized values on field forms.
- If the readings do not meet the stability criterion after extending the measurement period, record this difficulty in the field notes along with the fluctuation range and the median value of the last five or more readings.

5. For EWI or EDI measurements, proceed to the next station in the cross section and repeat steps 3 and 4. Record on field forms the mean (or median, if appropriate) value for each subsection measured.

6. When the measurement is complete, remove the sensor from the water, rinse it with deionized water, and store it.

7. Record the stream conductivity on the field forms:

- **In still water**—median of three or more sequential values.

- **EDI**—mean value of all subsections measured (use the median if measuring one vertical at the centroid of flow).
- **EWI**—mean or median of all subsections measured (see NFM 6.0).

Sub-sample measurement

Representative samples are to be collected and split or composited according to approved USGS methods. Measure the conductivity of samples as soon as possible after collection. If the sample cannot be analyzed immediately, fill a bottle to the top, close it tightly, and maintain the sample at stream temperature until measurement.

Reported conductivity values normally are determined on an unfiltered sample. Large concentrations of suspended sediment can be a source of measurement error—record such conditions in the field notes.

- If sediment concentrations are heavy, measure conductivity on both unfiltered and filtered sub-samples and record both values on the field form.
- If the conductivity value differs significantly between the filtered and unfiltered samples, report the filtered value as sample conductivity and identify it as a "filtered sample."

1. Calibrate the conductivity instrument system at the field site.

2. Select the sampling method (see NFM 6.0) and collect a representative sample.

3. Withdraw a homogenized sub-sample from a sample splitter or compositing device. Rinse the sample bottles three times with the sample—rinse them with sample filtrate, for filtered samples.

4. Rinse the conductivity sensor, the thermometer (liquid-in-glass or thermistor), and a container large enough to hold the dip-type sensor and the thermometer.

a. First, rinse the sensor, the thermometer, and the container three times with deionized water.

b. Next, rinse the sensor, the thermometer, and the container using sample water.

5. Allow the sensors to equilibrate to sample temperature, then discard the used sample water. Pour fresh sample water into a container holding the sensor and the thermometer. When using a dip-type sensor, do not let the sensor touch the bottom or sides of the measuring container.

6. Measure water temperature.

- If the conductivity sensor contains a calibrated thermistor, use this thermistor to measure water temperature.
- If the instrument is not temperature compensating, use a calibrated thermistor or a liquid-in-glass thermometer.
- Adjust the instrument to the sample temperature (if necessary) and remove the thermometer.

7. Measure conductivity.

a. Remove any air trapped in the sensor by agitating the sensor up and down under the water surface.

b. Read the instrument display.

c. Agitate the sensor up and down under the water surface, and read the display again.

d. Repeat the procedure until consecutive readings are the same.

8. Record the conductivity and the sample temperature on field forms.

- If the instrument is not temperature compensating, record the raw data and convert the values to conductivity at 25°C using temperature-correction factors provided by the manufacturer.
- Report the median of the readings to three significant figures on the field forms.
- Discard the sample into a waste container and dispose according to regulations.

9. Quality control-

- Repeat steps 3 through 8 with at least two fresh subsamples, rinsing the instruments once only with sample water.
- Subsample values should be within ±5 percent for conductivity ≤100 µS/cm, or ±3 percent for conductivity >100 µS/cm.
- If criteria cannot be met: filter the samples, report the median of 3 or more samples, and record this difficulty in field notes.

10. Rinse the sensor, the thermometer, and the container with deionized water. If another measurement is to be made within the next day or two, store the sensor in deionized water. Otherwise, store the sensor dry.

4.4 TURBIDITY METERS

Turbidity meters measure the amount of light scattered at right angles from a beam of light passing through the test sample. Turbidity readings are the measure of the interaction of light with suspended solid particles in the sample. Test results are read directly in Nephelometric Turbidity Units (NTUs) on an LCD digital readout.

4.4.1 Calibration

The turbidity meter is pre-calibrated in the factory, and a simple standardization is the only step required prior to testing.

4.4.2 Measurement

Turbidity measurements should be repeated three to five times to ensure accuracy and replication within the precision of the instrument.

Benchtop determination of turbidity is especially susceptible to negative bias from particle settling. Visually check for the presence of coarse material (sand or coarse silt) in the sample. Gently agitate the sample, then set it down. If particles rapidly settle to the

bottom (within 3-5 seconds), then coarse materials are present and the sample cannot be measured accurately using the static method. Static measurements made on such samples therefore must be coded to indicate that accuracy is qualified when being entered into a database.

Procedure*:*

Preliminary steps for benchtop turbidity determination:

1. Warm up the turbidimeter according to the manufacturer's instructions. Put on powderless laboratory gloves.

2. Rinse a clean, dry, scratch-free, index marked cell with a turbidity calibrant within the range of interest.

3. Gently agitate the calibrant, pour the calibrant into the sample cell to the fill mark, and dry the cell exterior with a lint-free cloth. When using a meter recently calibrated with an acceptable calibrant turbidity solution (formazin or styrene-divinylbenzene polymer), a verification calibrant may be used for this check measurement.

4. Follow the manufacturer's instructions for readout of turbidity value and record the turbidity of the calibrant used and the turbidity value measured in the calibration logbook. If readings are not within specifications for the indicated range, recalibrate the instrument for the turbidimeter using accepted calibration turbidity solutions.

For samples with turbidity less than 40 turbidity units:

1. Measure sample turbidity immediately or as soon as possible upon sample withdrawal.

    a. If discrete sub-samples are to be taken from a churn splitter or other sample-compositing device, remove samples for turbidity measurement along with other whole water samples. Avoid pouring the sample into a cuvette from a bottle, if possible. If not possible, then invert the bottle 25 times using a 1-second inversion cycle and pour off the sample immediately to capture suspended particles.

b. For drinking water, use an instrument that complies with EPA Method 180.1 or GLI Method 2. Measurements are reported in NTU or NTRU for EPA 180.1, or in FNMU for GLI Method 2.

2. Rinse a freshly cleaned cell with the sample to be tested.

3. For a discrete (static) sample, complete the following sequence of steps (through step 4a) without hesitation (skip to step 4 for flowthrough cell measurement).

a. Gently invert—do not shake—the sample 25 times to completely disperse the solids, taking care not to entrain air bubbles. Allow air bubbles to disappear before filling the sample cell.

b. Rapidly pour the sample into a sample cell to the line marked (to the neck if there is no line). Do not touch cell walls with fingers.

c. Remove condensation from the cell with a clean, soft, lint-free cloth or tissue. If condensation continues, apply a thin coating of silicon oil to the outside of the cell about every third time the cell is wiped dry of moisture. Allow samples to equilibrate to ambient temperature, if necessary, before sub-sampling to help minimize condensation problems. Note: warming the sample may change particle associations in the water matrix.

d. Before inserting the sample cell into the meter, ensure that no air bubbles are present in the cell. If necessary, degas the sample according to the manufacturer's instructions. Air bubbles can cause significant positive bias in turbidity measurements.

e. Orient the calibration cell in the cell holder according to the index marks—the calibration cell and sample cell must have identical orientation when in the instrument measurement chamber.

4. Determine the measured turbidity value of the sample directly from the instrument scale or by using the instrument value and calibration curve, as is appropriate for the

instrument being used. For samples with less than 1 turbidity unit, see the Technical Note under step 4d.

a. Record the very first readings after placement of the sample cell in the measurement chamber. If readings are unstable, then particle settling may be occurring. If so, gently re-invert the cell 25 times and record at least three readings over a short, defined time interval (for example, 30 seconds to 1 minute).

b. Repeat at least twice with fresh sample, until three or more sample values fall within ±10 percent.

c. Samples that contain significant color should be diluted if using EPA Method 180.1 (for samples with turbidity greater than 40 units see below "For samples, including drinking water, with turbidity greater than 40 turbidity units," step 3). Results of diluted samples must be qualified with a “d” in the “Value Qualifier Code” field for data entered into the USGS NWIS database.

d. Report the median of the three or more sequential readings that fall within ±10 percent.

Technical Note: When using low-level reporting scales, you may need to subtract a correction factor from the reading to correct for stray light. For example, the Hach Company reports the correction for the 0.2-NTU scale to be on the order of 0.04 NTU for the Hach 2100P. The stray-light correction is determined by reading turbidity from an empty instrument (without cuvette).

5. Record the data. If particle settling or instability in initial readings was a problem, add documentation to field notes in the log book.

For samples, including drinking water, with turbidity greater than 40 turbidity units:

1. Select an appropriate instrument.

- For drinking water, use EPA Method 180.1, a compliant instrument, and NTU or NTRU reporting units; alternatively, select the GLI Method 2, a compliant instrument, and FNMU reporting units. Reporting units for these methods must be remarked with an "E" code in NWIS for turbidities greater than 40.
- For study objectives other than drinking water, choose instruments according to study objectives.

2. Obtain a discrete sample.

- For drinking-water samples, proceed to step 3.
- For non-drinking-water samples, skip to step 4.

3. For drinking-water samples, dilution is required to comply with USEPA regulations.

a. Dilute the sample with one or more equal volumes of turbidity-free water until turbidity is less than 40 turbidity units after mixing and degassing.

b. Record the volume of turbidity-free water used for dilution. Follow steps 1-5 from the previous section for samples with turbidity less than 40 turbidity units.

c. Skip to step 5, below

4. For non-drinking-water samples (where USEPA compliance is not required), with 100 and 1,000 turbidity-unit ranges only — place a cell riser (if available) into the cell holder before inserting the sample cell. This decreases the length of the light path in order to improve the linearity of measurements. Do not use the cell riser for the lower turbidity ranges.

a. For turbidimeters with adjustable ranges and signal-processing capabilities (for instance, ratio mode to compensate for high particle densities), select the desired configuration and operate according to manufacturer's recommendations. Some instruments will automatically switch to different modes (for example, ratio mode) or to a different light source. Record instrument mode on field sheets.

b. Select the desired range on the turbidimeter.

5. Fill the cell with sample water:

a. Hold the cell by the rim (top lip), not beneath the lip.

b. Gently agitate the sample 25 times. Without hesitation, carefully but rapidly pour sample water into the cell to the fill mark.

c. Wipe the exterior of the cell using a soft, lint-free cloth or tissue to remove moisture (condensation) from cell walls.

d. If necessary, apply a thin layer of silicon oil onto the exterior of the cell to reduce condensation on the cell and mask slight scratches and nicks.

e. If rapid particle settling is occurring, steadily invert the cell 25 times, taking care not to shake too vigorously, which could entrain gases in the sample.

6. Record the sample turbidity.

Most modern turbidimeters will adjust initial sample readings directly into a final reading based on the previous calibration. If the meter does not have this capability, you will need to read values from a calibration curve constructed previously.

a. Record the very first readings after placement of the sample cell in the measurement chamber. If readings are unstable, particle settling may be occurring: gently re-invert the cell 25 times and record at least three readings over a defined time interval (for example, 30 seconds to 1 minute).

b. Repeat at least twice with fresh sample until three or more sample values fall within ±10 percent.

c. Samples that contain significant color should be diluted if using EPA Method 180.1. Results of diluted samples must be qualified with a “d” in the “Value Qualifier Code” field for data entered into the USGS NWIS database.

d. Report the median of the three or more sequential readings that fall within ±10 percent.

- For diluted water samples, the measured turbidity must be converted based on the amount of dilution, according to the following equation:

$$T_s = T_d \times (V_o+V_s)/V_s$$

where *T*s= turbidity of the environmental sample, *T*d=turbidity of the diluted sample, *V*o = volume of turbidity-free water in the diluted mixture, and *V*s= volume of the environmental sample in the diluted mixture.

EXAMPLE: If five volumes of turbidity-free water were added to one volume of sample, and the diluted sample showed a turbidity of 30 units, then the turbidity of the original sample is computed as 180 units.

e. Report turbidity as follows, using method codes as described in http://water.usgs.gov/owq/turbidity_codes.xls:

- For EPA Method 180.1, use NTU or NTRU.
- For GLI Method 2, use FNMU.
- For non-diluted, non-USEPA-compliant measurements, use the appropriate reporting units.

4.5 DISSOLVED OXYGEN METERS

Dissolved oxygen (DO) meters measure the quantity of oxygen dissolved in water. In a typical DO meter, the tip of the probe consists of a cell enclosed by a selective membrane in a protective holder containing the electrolyte and electrodes.

4.5.1 Calibration

Always calibrate the instrument according to the manufacturer's specifications. For an accurate calibration, the probe may require immersion in water in an airtight container. If an open container is used for calibration, the margin of error is approximately 0.1 ppm. If the calibration is performed above sea level, a correction will be made for the difference in altitude. Certain table listings are available for oxygen solubility as a function of temperature and salinity. Refer to the manufacturer's specifications regarding slope calibration.

Instrument systems for the amperometric or the luminescent-sensor methods must be properly calibrated and tested before each field trip and cleaned in the field after each use.

- *Amperometric instruments*

  Different manufacturers recommend different calibration frequencies for membrane-electrode DO meters; however, virtually all state that optimum instrument performance and data quality will be obtained by frequent calibration. Calibration and operation procedures for the amperometric method differ among instrument types and makes—refer to the manufacturer's instructions.

- *Luminescent-sensor instruments*

  Luminescent-based sensors are pre-calibrated by the manufacturer and most manufacturers' literature suggests that no further calibration is warranted. The accuracy of factory calibrations, however, may not satisfy the data-quality objectives of a specific program. Frequency of calibration can have a significant effect on the overall accuracy and precision of DO measurements; therefore, users of these meters are advised to make frequent calibration checks and to recalibrate as frequently as required to meet specific data-quality objectives.

- *One-point and two-point calibrations*

  Calibration for most amperometric DO instruments and some luminescent-sensor instruments can only be checked with a 1-point calibration at 100-percent saturation. For these instruments, a zero DO check should be performed routinely as an evaluation of sensor performance. Because the sensors on DO instruments may be slow to respond after the zero check, the sensor should be thoroughly rinsed with deionized water before use.

Some instruments allow for 2-point calibrations at 0-percent and 100-percent saturation. Follow the manufacturer's instructions for those instruments with 2-point calibration functionality. Verifying instrument performance at zero DO and using a 2-point calibration can be particularly important for data accuracy when the instrument will be used to measure low DO concentrations (less than 5 mg/L).

- *Correction for atmospheric pressure*

Atmospheric pressure, the temperature of the water or water vapor, and the conductivity (or salinity) of the water must be known to determine the theoretical amount of oxygen that can be dissolved in water. Record all calibration information in instrument log books and copy calibration data onto field forms at the time of calibration.

Ambient atmospheric pressure is true atmospheric pressure at the measurement site, not that which has been adjusted to sea level. Atmospheric pressure reported by the National Weather Service generally is not the true (ambient) value. Weather Service atmospheric readings usually are adjusted to sea level and must be adjusted back to the elevation of the weather station. Upon request, a weather station may provide ambient atmospheric pressure.

- Use a calibration-checked pocket altimeter-barometer to determine ambient atmospheric pressure to the nearest 1 millimeter (mm) of mercury.
- Check the accuracy of all field barometers before each field trip, and record readings and adjustments in the log book. If possible, check barometer accuracy with information from an official weather station.
- Use Table 1 and Figure 1 if the value used for atmospheric pressure has been adjusted to sea level.
- To correct weather station readings adjusted to sea level to ambient atmospheric pressure: subtract appropriate values shown (Table 1, Figure 1) from atmospheric readings adjusted to sea level (shown in millimeters of mercury).

Although atmospheric pressure does not decrease linearly with increases in elevation, linear interpolation is acceptable within the elevation ranges given in Table 1. Alternatively, plot the values from Table 1 and extrapolate subtraction factors directly from the graph (Figure 1). Many instruments have the pressure-temperature algorithm stored in internal memory. Interactive tables also are available for user-specified temperature, pressure, and salinity at http://water.usgs.gov/software/dotables.html.

Table 1. Factors used to correct atmospheric pressures adjusted at sea level NGVD, National Geodetic Vertical Datum of 1929

Elevation of weather station (in feet, NGVD)	Value to subtract (mm of Hg)
0	0
1000	27
2000	53
3000	79
4000	104
5000	128
6000	151

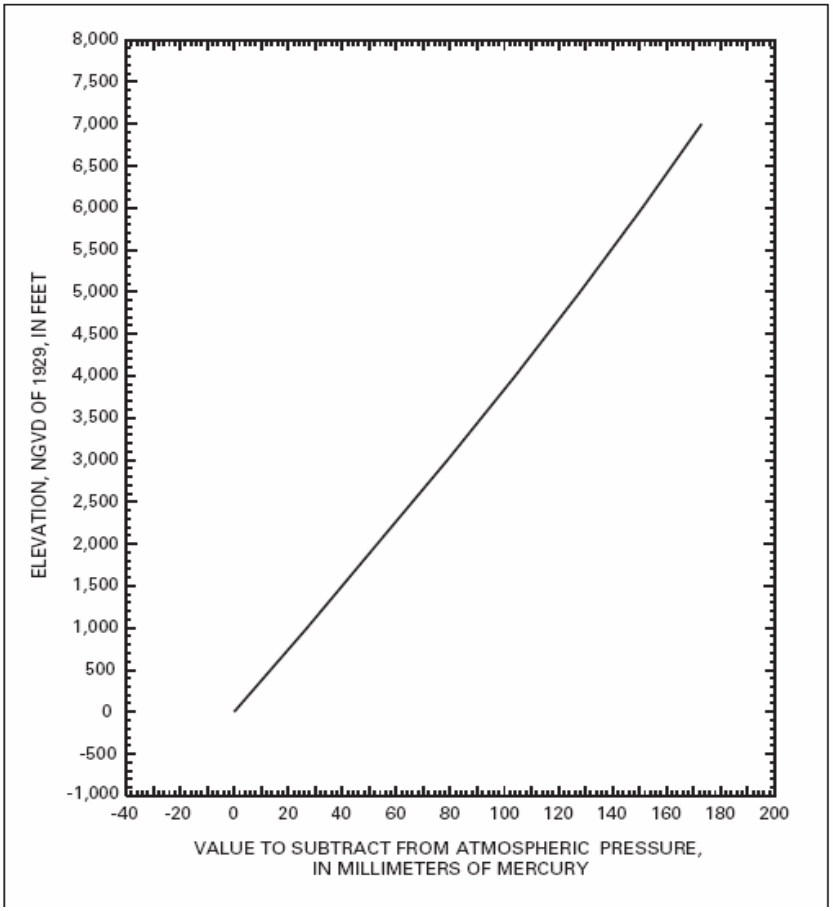

Figure 1. Factors used to correct atmospheric pressures adjusted to sea level.

Procedure 1—Air-calibration chamber in air

This calibration method is most commonly recommended by manufacturers of amperometric instruments. Calibration chambers are either built into the instrument case or are provided as separate components by the manufacturer. Use the calibration chamber provided or recommended by the manufacturer.

1. Wet the inside of the calibration chamber with water. Then pour out the water (but leave a few drops). Remove any water droplets on the sensor membrane and insert the sensor into the chamber (this ensures 100-percent humidity).

2. If using an amperometric instrument, allow 10 to 15 minutes for the DO sensor and the air inside the calibration chamber to equilibrate.

3. Using your calibration pocket altimeter-barometer, read the ambient atmospheric pressure checked to the nearest 1 mm of mercury.

4. Measure the temperature in the calibration chamber and observe the readings until the instrument stabilizes. Read the temperature to the nearest 0.1^{o}C. The temperature inside the chamber should approximate the water temperature, measured with a calibrated thermometer.

> **Technical Note for Amperometric Instruments:** Most instrument manufacturers recommend calibrating at temperatures that are at least within 10^{o}C of the ambient water temperature. The most accurate calibration will be achieved if the temperature difference between the environmental water and the calibration chamber is minimized as much as possible.

5. Use Table1 to determine the DO saturation at the measured temperature and atmospheric pressure.

6. Following the manufacturer's instructions, adjust the calibration control until the instrument reads the DO saturation value determined from Table 1. Verify that the instrument reading is within ±0.2 mg/L of the computed saturation value, or use more stringent accuracy criteria that reflect the data-quality requirements of the study. The luminescent-sensor instrument is now calibrated and ready for use.

7. When working with an amperometric instrument, remove the sensor from the calibration chamber and check to see if any water droplets are on the membrane. Water droplets on the membrane cause improper calibration. If water droplets are present, recalibrate the instrument; otherwise the instrument is now calibrated and ready for use.

Procedure 2—Calibration with air-saturated water

In this procedure, the DO sensor or instrument system is calibrated against water that is saturated with oxygen at a known temperature and ambient atmospheric pressure.

1. The temperature of water used for calibration should be about the same as the temperature and conductivity of the water to be measured.

- **If working at the field site—**obtain about 1 liter (L) of water from the water body to be measured.
- **If working in the laboratory—**obtain about 1 L of deionized water or tap water.

2. Place the DO sensor and calibration water in a large beaker or open-mouth container. (Some manufacturers supply an air-saturated water-calibration vessel.)

- Allow the sensor to come to thermal equilibrium with the water temperature.
- Shield the beaker or container from direct sunlight and wind to minimize temperature variations.

3. Aerate the water for 5 to 10 minutes. Using a battery-operated aquarium pump or minnow-bucket aerator and a short piece of tubing, attach a gas diffusion stone to the end of the tubing and place it at the bottom of the beaker of calibration water. Avoid placing the instrument in the stream of air bubbles.

4. Determine if the water is 100-percent saturated with oxygen.

- Observe the instrument reading while aerating the calibration water.
- When no change in the DO reading is observed on the instrument for 4 to 5 minutes, assume that the water is saturated.

5. Using your pocket altimeter-barometer, read the ambient atmospheric pressure to the nearest 1 mm of mercury.

6. Read the temperature of the calibration water to the nearest 0.1°C.

7. Using oxygen solubility Table 1, determine the DO saturation value at the measured temperature and atmospheric pressure of the calibration water.

*****Skip to Step 9 if using an amperometric instrument*****

8. **For luminescent-sensor instruments:** Following the manufacturer's instrument calibration instructions, verify that the instrument reading is within ±0.2 mg/L of the computed saturation value. (Alternatively, apply a more stringent accuracy criterion that reflects study data-quality requirements.) **The luminescent-sensor instrument is now calibrated and ready for use.**

9. **For amperometric instruments:** Adequate flow of water across the surface of the membrane is required for accurate measurements. Recommendations for flow velocity vary by manufacturer, with most recommending about 1 foot per second (ft/s).

- Provide suitable turbulence in the air-saturated water by physical or mechanical means to maintain the required flow rate past the membrane, avoiding the creation of air bubbles at the water-sensor interface.
- Maintain this flow rate when making measurements and adjusting instrument calibration.

10. **For amperometric instruments**: Turn off the aerator and take care to prevent any air bubbles from adhering to the membrane. Following the manufacturer's instructions, set or adjust the calibration control until the instrument reads a saturation value of DO as determined above. Verify that the instrument reading is within ±0.2 mg/L of the computed saturation value, or use more stringent accuracy criteria that reflect the data-quality objectives of the study.

Procedure 3—Air-calibration chamber in water

This calibration method is applicable only to amperometric instruments. An air-calibration chamber permits calibration of the DO sensor at the temperature of the water in which the DO concentration is to be measured. This calibration procedure minimizes errors caused by temperature differences. Air-calibration chambers for in-water

calibrations currently are not available on the open market and one of the most common, the YSI 5075A calibration chamber, is no longer manufactured. For most multi-parameter water-quality instruments, the manufacturer-provided ground-water flow cell may be modified and used as an air-calibration chamber in water. The modification requires the cell to be mounted on the sonde with one port of the cell plugged and the other port vented to the atmosphere with tubing.

1. Insert the sensor probe into the rings of the DO wand and dip this calibration chamber into the surface or ground water to be measured, allowing the temperature readings to stabilize. Remove the wand and pour out the excess water, leaving a few drops.

- Check for and remove any water droplets on the sensor membrane.
- Insert the DO sensor into the wet chamber (this ensures 100 percent humidity).
- If a YSI model 5739 sensor is used, the pressure-compensating diaphragm on the side of the sensor must be enclosed within the calibration chamber during calibration.
- Check that no water can leak into the calibration chamber and that the membrane does not have droplets of water adhering to it. The water droplets reduce the rate of oxygen diffusion through a membrane, producing erroneous results.

2. Immerse the calibration chamber into the water to be measured. Allow 10 to 15 minutes for the air temperature inside the chamber to equilibrate with the water (see the **Technical Note** in Procedure 1).

- For streams, choose an area of the stream that closely approximates mean stream temperature. In shallow streams, try to place the chamber in an area that represents the stream but that is shaded from direct sunlight.
- For ground water, use temperature-stabilized purge water or other clean water having a temperature that closely approximates that of the ground water.

3. Using a calibration-checked pocket altimeter-barometer, determine the ambient atmospheric pressure to the nearest 1 mm of mercury.

4. Read the temperature within the chamber to the nearest 0.1°C, using a calibrated thermometer.

- The temperature inside the chamber should approximate the water temperature.
- If the two temperatures do not match, allow additional time for equilibration of the chamber with the water temperature.
- If the temperature of the chamber still does not approximate the water temperature, the thermistor in the DO sensor might be malfunctioning. Compare water temperature measured by the DO meter and a calibrated field thermometer. If the two measurements vary by more than ±0.2°C, the calibration should be discontinued and the DO meter thermistor should be repaired following the manufacturer's recommendations.

  Technical Note: Most instrument manufacturers recommend calibrating at temperatures that are at least within 10°C of the ambient water temperature. The most accurate calibration will be achieved if the temperature difference between the environmental water and the calibration chamber is minimized as much as possible.

5. Use Table 1 to determine the DO saturation value at the measured water temperature and atmospheric pressure.

6. Following the manufacturer's instructions, set or adjust the calibration control until the instrument reads a DO saturation value determined from oxygen solubility (Table 1). Verify that the instrument reading is within 0.2 mg/L of the computed saturation value, or use more stringent accuracy criteria per the data-quality objectives of the study. The instrument is now calibrated and ready for use. Remove the sensor from the calibration chamber.

4.5.2 Measurement

The solubility of oxygen in water depends on the partial pressure of oxygen in air, the temperature of the water, and the dissolved-solids content of the water.

Standard determinations of dissolved oxygen in surface water represent the cross-sectional median or mean concentration of dissolved oxygen at the time of observation.

Measuring the DO concentration at one distinct spot in a cross section is valid only for flowing water with a cross-sectional DO variation of less than 0.5 mg/L. Discerning such variation requires a cursory cross-section measurement. The effort involved in collecting this cross-section information is only slightly less than making an equal-width-increment (EWI), equal-discharge-increment (EDI), or multiple-vertical cross-sectional measurement. Measurements made at multiple locations in the cross section are recommended when possible.

- Determining DO for a single vertical at the centroid of flow at the midpoint of the vertical only represents the cross section under ideal mixing conditions.
- Do not measure DO in or directly below sections with turbulent flow, in still water, or from the bank, unless these conditions represent most of the reach or are required by the study objectives.
- Apply a salinity correction to the saturation values after the DO measurement, if needed

Dissolved oxygen must be measured in situ. Never measure DO from a sample splitter.

Procedure:

1. Calibrate the DO instrument at the field site and check that the temperature thermistor has been certified by the USGS Water Science Center within the past 4 months
2. Record the DO variation from the cross-sectional profile and select the sampling method
   - **Flowing, shallow stream**—Wade to the location(s) where DO is to be measured.
   - **Stream too deep or swift to wade**—Lower a weighted DO sensor with a calibrated temperature sensor from a bridge, cableway, or boat. (Do not attach the weight directly to the sensors or sensor cables, because this could damage the sensors or sensor cables.)
   - **Still-water conditions**—Measure DO at multiple depths at several points in the cross section.
3. Immerse the DO and temperature sensors directly into the water body and allow the sensors to equilibrate to the water temperature (no less than 60 seconds).

Notes for amperometric instruments only:

If the water velocity at the point of measurement is less than about 1 ft/s, use a stirring device or stir by hand to increase the velocity. (To hand stir, raise and lower the sensor at a rate of about 1 ft/s, but do not break the surface of the water.) The stir-by-hand method may not be appropriate in lakes, reservoirs, or slow-moving waters (for example, bayous) as these water bodies may be stratified at the point of measurement, making accurate DO measurements impossible. This could be especially problematic in areas where DO

concentrations change substantially over short distances, such as near the thermocline or bottom sediments.

- High stream velocity can cause erroneous DO measurements.

4. Record the temperature without removing the sensor from the water.

5. After the instrument reading has stabilized, record the median DO concentration

The reading should stabilize to within ±0.2 mg/L.

6. For EWI, EDI, or multiple-vertical measurements, proceed to the next station in the cross section and repeat steps 3 through 5. When measurements for the stream have been completed, remove the sensor from the water, rinse it with deionized water, and store it according to the manufacturer's instructions.

7. Record DO concentrations on the field forms:

- **In still water**—median of three or more sequential values.
- **EDI**—mean value of all subsections measured (use the median if measuring one vertical at the centroid of flow).
- **EWI—mean (or median)** of all subsections measured.

5.0 QUALITY ASSURANCE AND QUALITY CONTROL

All equipment calibration data must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP or manufacturer's recommendations. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and other procedures referenced herein. All equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

6.0 DECONTAMINATION

All equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated in the calibration process shall be managed in accordance with the procedures outlined in SOP-12.

7.0 REFERENCES

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Date: January 31, 2008

OU3 SOP 11 (Rev 1)

Title: GPS DATA COLLECTION

APPROVALS:

EPA Remedial Project Manager — Date: 3/20/08

SOP Author — Date: 3/20/2008

Revision Number	Date	Reason for Revision
0	9/26/07	
1	1/31/08	Update based on field team input

LIBBY ASBESTOS SUPERFUND SITE OU3

STANDARD OPERATING PROCEDURE No. 11

GPS DATA COLLECTION

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is a general guidance document for the collection of coordinates of point locations using a Global Positioning System (GPS) unit. The GPS is a worldwide, satellite-based system with location positioning capabilities. The system is administered and managed by the Department of Defense. It is comprised of:

- a space segment of approximately 24 operational satellites in complimentary orbit,
- a ground control segment made up of a network of control stations around the globe, and
- a user segment, which includes anyone who uses GPS to collect locational information.

The system utilizes precise time and radio signals to determine distances from satellites to user GPS receivers. Distances are most commonly calculated by using the time it takes for a radio signal code to be transmitted from the satellite and received by the GPS unit. Precise time is critical to the successful operation of the system. The control stations ensure that the satellites employ synchronized, atomic clock-derived universal time coordinates (UTC), commonly known as Greenwich Mean Time (GMT). Receiver units collecting four satellite signals can determine the geodetic (x, y, z) location through a process of mathematical triangulation. The satellite signals contain precise time and satellite position information.

GPS technology is used as a method of accurately determining the coordinates of point locations. The three-dimensional position, or the x, y, and z geodetic coordinates, are determined for the point locations; however, only the x and y values are primarily used. This is due to the processes involved in the system; the vertical GPS coordinates are approximately

half as accurate as the horizontal GPS coordinates. The position reported by the GPS unit is based on the geodetic model selected. The vertical, or z coordinate, value is not as accurate as the reported position due to the geometry of the satellite constellation relative to the receiver's position on the earth.

GPS is one of the arrays of tools for accurately determining location in the field. The collection of x, y, and z coordinates (for gross data collection) for locations in the field using GPS is useful for a variety of purposes, including accurate sample locations, locational correlation of remotely sensed data with ground truth locations, and efficiently collecting better spatial data.

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects project-specific field documentation with input from other key project staff, and appropriate oversite agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Field Technician (or other designated personnel): Assists the FTL and/or field geologist, hydrogeologist, or engineer in the implementation of field tasks and field documentation.

4.0 PROCEDURES

There are three major types of GPS units available: survey-grade units, mapping-grade units, and recreational-grade units. For the collection of environmental/natural resource data, mapping-grade and recreational-grade units are usually sufficient. The type of GPS unit employed should meet the data collection needs as outlined in the project work plan.

Recreational-grade GPS units can be used to acquire location information (generally points) when spatial accuracy is not paramount to the project. Recreational GPS units do not have data dictionaries for storing attribute information with the point location. The procedures described here are geared toward hand-held recreational GPS units. Consult the specific instrument's instruction manual for details on operation.

There has been, and will continue to be, a considerable and rapid evolution in GPS techniques and technologies. Adjustments to the following operational procedures may be necessary to reflect these rapid changes in technology.

4.1 Method

Planning

If a recreational-grade GPS meets the criteria of the project, the unit chosen must have the capability of downloading collected data to a personal computer. This is usually accomplished with a parallel or USB cable connection.

Much of the data collected by GPS will eventually reside in a relational database. Each GPS feature collected should contain a unique identifier that relates the feature to an associated record in a database. Since recreational GPS units have only one text field for input, careful consideration should be given to the use of this field and the design of unique identifiers.

Data Collection

Locational data are captured by recreational-grade GPS units as *waypoints*. When taking a waypoint, enter the Location ID in the text field provided. It is also recommended that reference points be collected occasionally. These reference point positions should be taken at known

locations (e.g., site headquarters office, stream confluences) which can later be used in GIS to QC the accuracy of waypoint data.

If navigation to preset waypoints is applicable to a project, they must be loaded onto the GPS unit before departure to the field. It is also recommended to have printed topographic maps of the waypoint locations in order to maximize field time and efficiently navigate between waypoints.

Data Processing

GPS units should be downloaded once a day or after each field session. Data should be downloaded both as a text file and a shapefile. Points should be checked for reasonable spatial accuracy and errors. Subsequent downloads should be error-checked in the same manner. When data collection is finished, all files should be compiled into one spatial file.

Additional Documentation

Regardless of the type of GPS unit used to collect locational data, all resulting GIS datasets need to have information documenting how the GPS data were collected. Documentation can be recorded at the time of data point collection and/or can be stored along side the electronic data set with a simple readme text file.

The following details are suggested as items to include in data collection:

- Name of project
- Name(s) of data collectors
- Coordinate system (projection, datum & zone)
- Type (or types) of GPS units used
- The range of field collection dates

Below is an example of typical GPS data collection using a hand-held recreational-use GPS device. It serves only as an example of data collection and is not intended to provide detailed step-by-step instructions operation. Always refer to the owner's manual for specific instructions on device operation and data collection.

- Turn GPS unit on by pressing and releasing the power key.

- Following the Welcome Page the Satellite Page will be next.

- After sufficient satellites have been acquired, change to Position Page.

- When locating a sample location, use the sample identification as described in the field sampling plan.

- To record a location press the Mark key; the longitude, latitude, time, and date will be saved. Record the information into a field log book, and save the information in the GPS unit with a unique identification name and/or number to be downloaded later.

- Then enter OK to return to the Position Page.

- To turn off the GPS unit press and hold down the power key.

5.0 QUALITY ASSURANCE AND QUALITY CONTROL

Only data with high geometrical strength or low percent dilution of position (PDOP) will be used to ensure high accuracy. The Field Team Leader or designated QA reviewer will check and verify that the GPS coordinates are collected using the appropriate Datum, are entered into a field logbook or electronic database on a daily basis and that coordinates entered into project records match those recorded in the GPS-unit memory. If any corrections are necessary, the field team lead or other field personnel will make those corrections before coordinates are transmitted to data users.

All GPS equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

6.0 REFERENCES

U.S Environmental Protection Office of Environmental Information. *Global Positioning Systems-Technical Implementation Guidence.*September 2003. Available online at http://www.epa.gov/OEI/pdf/GPS-TIG.pdf

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Garmin GPSmap 60CSx Operators Manual

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Date: September 26, 2007 OU3 SOP 12 (Rev. 0)

Title: INVESTIGATION DERIVED WASTE (IDW) MANAGEMENT

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		9/26/07
SOP Author		9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	--

1.0 INTRODUCTION

This SOP is based on MWH SOP-07, Investigation-Derived Waste (IDW) Management, Revision 1.0, April 2007, modified for use at the Libby Asbestos Superfund Site OU3. IDW may be generated during field investigations at the Libby Asbestos Superfund Site OU3. The National Contingency Plan (NCP), codified in 40 Code of Federal Regulations (CFR) 300, requires that IDW be handled to attain all the applicable or relevant and appropriate requirements (ARARs) to the extent practicable, considering the urgency of the situation. The purpose of this SOP is to present procedures to be followed in the management of IDW generated during the field investigations.

Typical IDW generated during field activities are solid wastes and may include (but are not limited to) the following media and waste types:

Fluids	Solids
Purge water and groundwater	Soils and soil cuttings
Drilling mud	Plastic tarps or sheeting
Grout	Drill pipe and well casing/screen
Decontamination fluids and wastewater	Decontamination solids
	Disposable equipment (i.e., rope, bailers, sampling equipment, & other consumables)
	Spent personal protective equipment (PPE)
	Used containers, sample bottles
	Packaging materials

The above wastes may or may not be encountered, generated or managed while performing field investigations. However, all solid waste streams will be characterized to determine if they are hazardous wastes per 40 CFR § 262.11 for the purposes of handling and disposal. Guidance from this document shall be used as part of project planning to estimate total volumes of IDW likely to be generated as well as how the IDW will be managed and disposed.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in IDW handling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Area of Contamination (AOC) unit: The AOC unit concept is critical to the IDW management at a CERCLA investigation site. Although EPA has not promulgated a definition of an AOC unit, an AOC unit is generally an area within a CERCLA investigation site with similar characteristics with respect to contamination and the associated risks to human health and the environment. A CERCLA investigation site may contain one or more AOC units.

Decontamination fluids: Any fluids, including aqueous wash water, solvents, and contaminants that are used or generated during decontamination procedures.

Decontamination solids: Any solids, including soils and soil cuttings, fill materials, and contaminants that are generated during decontamination procedures.

Grout: A fluid mixture of cement and water (neat cement) of a consistency that can be forced through a pipe and placed as required.

Hazardous waste: A solid waste that meets the definition of a hazardous waste under RCRA as defined in 40 CFR § 261.3.

Investigation-derived waste (IDW): Solid wastes, as defined in 40 CFR § 261.2, directly generated as result of performing the field activities.

Nonhazardous waste: A solid waste that does not meet the definition of a hazardous waste as defined in 40 CFR § 261.3 or is excluded from hazardous waste regulation per 40 CFR § 261.4(b).

Soils and soil cuttings: Solid material generated from excavation or drilling processes. Soils may include native soils, fill materials, and/or other historical plant waste streams used as fill materials on the site.

Solid waste: Any waste stream (solid, liquid or containerized gas) that meets the definition of solid waste under RCRA as defined in 40 CFR § 261.2**.**

4.0 RESPONSIBILITIES

This section presents a brief definition of the field team roles and responsibilities for management of IDW generated while conducting field investigations. This list is not intended to be a comprehensive list as additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Responsible to ensure that all field team members are properly trained per their responsibilities associated with IDW and that appropriate equipment and facilities are available for appropriate IDW management.

Field Team Leader (FTL): Implements the field program and supervises all field team members in the appropriate management of IDW. Ensures that only properly trained personnel are managing IDW on the site.

Environmental, Health and Safety (EHS) Officer: Assists the Team Leader in the supervision of all IDW management on site. The EHS officer shall be responsible for all IDW identification

and characterization, on site disposal, off site shipment and disposal, waste accumulation, emergency response and contingency planning, IDW training, and IDW reporting and recordkeeping.

Project Team Members: Ensure that they are properly trained prior to any IDW management as well as follow the appropriate IDW procedures and training.

5.0 REGULATORY BASIS AND GUIDANCE

IDW encountered, generated, or managed during the field investigations may contain hazardous substances as defined by CERCLA. Some IDW may be hazardous wastes under RCRA while others may be regulated under other federal laws such as TSCA. These regulatory requirements may be applicable or relevant and appropriate requirements (ARARs) which impact how the IDW is managed.

5.1 EPA Guidance on IDW Management

The management of IDW shall be in accordance with EPA Guidance "Management of Investigation-Derived Wastes During Site Inspections", May 1991 (EPA, 1991). The specific elements of EPA's guidance for IDW management are as follows:

- Characterizing IDW through the use of existing information (manifests, MSDSs, previous test results, knowledge of the waste generation process, and other relevant records) and best professional judgement.

- Delineating an AOC unit for leaving RCRA hazardous soil cuttings within the unit.

- Containerizing and disposing of RCRA hazardous groundwater, decontamination fluids, PPE, and disposable equipment at RCRA Subtitle C facilities.

- Leaving on-site RCRA nonhazardous soil cuttings, groundwater, and decontamination fluids preferably without containerization and testing.

In general, EPA does not recommend removal of wastes from sites, in particular, from those sites where IDW do not pose any immediate threat to human health or the environment. Actions taken during field investigations with respect to IDW that leave conditions essentially unchanged should not require a detailed analysis of ARARs or assurance that conditions at the site after field investigations will comply with the ARARs. At the same time, field personnel ensure that their handling of IDW does not create additional hazards at the site.

In brief, compliance with the NCP can generally be assured by:

1) Identifying contaminants, if any, present in the IDW based upon existing information and best professional judgement; testing is not required in most circumstances.

2) Determining ARARs and the extent to which it is practicable to comply with them.

3) Delineating an AOC unit based upon existing information and visual observation if soil cuttings are RCRA hazardous.

4) Burying RCRA hazardous soil cuttings within the AOC unit, so long as no increased hazard to human health and the environment will be created. Containerization and testing are not required.

5) Containerizing RCRA hazardous groundwater and other RCRA hazardous IDW such as PPE, disposable sampling equipment, and decontamination fluids for off-site disposal.

5.2 Hazardous Waste Regulation

The RCRA hazardous waste regulations are clearly ARARs for hazardous IDW generated and managed during field investigations. However, with the application of EPA IDW guidance, RCRA requirements apply to management of IDW in the following manner:

- If RCRA hazardous IDW is stored or disposed off-site, then comply with all RCRA (and other ARAR) requirements.

- If RCRA hazardous IDW is stored on-site, then comply with RCRA (and other ARAR) requirements to the extent practicable.

For these field investigations, the following general guidance is expected to be practicable and therefore followed, recognizing that each situation will be evaluated against EPA IDW guidance (EPA, 1991) as well as RCRA hazardous waste requirements and other ARARs:

- IDW may be assumed not to be a "listed" hazardous waste under RCRA 40 CFR 261 Subpart D, unless available information about the site suggests otherwise.

- IDW characterization to determine if the IDW exhibits RCRA hazardous waste characteristics do not typically require testing if the characterization can be made by "applying knowledge of the hazardous characteristics in light of the materials or processes used" or by historical testing consistent with 40 CFR § 262.11(c).

- Compliance with the RCRA hazardous waste generator requirements of 40 CFR Part 262 for all RCRA hazardous IDW generated and/or managed (with exception of soil cuttings managed in accordance with the EPA IDW guidance). It is presumed that the RCRA hazardous IDW generated will fall within the large quantity generator (LQG) requirements.

- Land disposal does <u>not</u> occur (and thus the Land Disposal Restrictions [LDR] of 40 CFR Part 268 are <u>not</u> applicable) when IDW soil cutting wastes are:

  - Moved, stored or left in place within a single AOC unit;
  - Capped in place;
  - Treated in situ (without moving the IDW to another AOC unit for treatment); or
  - Processed within the AOC unit to improve structural stability (without placing the IDW into another AOC unit for processing).

- Conversely, land disposal does occur (and the LDR of 40 CFR Part 268 are applicable) when IDW soil cutting wastes are:

  - Moved from one AOC unit to another AOC unit for disposal;
  - Moved outside an AOC unit for treatment or storage and returned to the same AOC unit for disposal;
  - Excavated from an AOC unit and placed in a container, tank, surface impoundment, etc. and then re-deposited back into the same AOC.

5.3 TSCA PCB Regulation

IDW containing PCBs at detectable levels may be generated, although the concentration of PCBs in any IDW generated is expected to be far below 50 ppm. However, IDW generated will be evaluated for PCBs and managed according to the following per the requirements of 40 CFR Part 761 Subpart D:

- Liquid IDW at concentrations greater than or equal to 50 ppm PCBs will be incinerated off-site at a TSCA-approved incinerator site.

- Nonliquid IDW at concentration greater than or equal to 50 ppm PCBs may be incinerated, treated by an equivalent TSCA-approved method, or disposed in a TSCA chemical landfill off-site.

- IDW at concentrations less than 50 ppm are generally not regulated under TSCA, and may be disposed in an acceptable Subtitle D facility.

6.0 DESCRIPTION OF ANTICIPATED IDW MANAGEMENT

The following subsections provide a description of the anticipated IDW to be encountered, generated, and/or managed at the Libby Asbestos Superfund Site OU3 during field activities and the anticipated management of each. It should be noted that this information is provided for planning purposes, and will be evaluated and may need to be revised based upon actual experience while on site.

6.1 Soil and Soil Cuttings

During field investigations, surface soil samples, samples of mine waste rock, and samples of fine tailings will be collected. Only a small portion of material will be collected for analysis. While the soil and soil cuttings IDW generated will be evaluated on a case-by-case basis, the general approach will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Characterizing the IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the waste generation process, and other relevant records) and best professional judgement.

- Soil and soil cuttings which are not used directly for sample makeup will not be taken outside of the AOC unit in which they were generated.

- Soil and soil cuttings within the AOC where they are generated will be placed back into the same investigation pit, trench, or bore hole and in the same order from which the material was removed, to the extent practicable and unless noted otherwise in the FSP.

- Soil cuttings potentially requiring RCRA disposal will be handled per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

6.2 Spent Sampling-Related Equipment

During field investigations, spent sampling-related equipment will be generated. This may include (but not limited to) plastic sheeting/tarps, rope, bailers, sampling equipment, spent PPE, sample bottles, used containers, packaging materials, and other consumables. Although the vast majority of the spent sampling-related equipment is expected to be nonhazardous, these IDW may contain a listed hazardous waste (e.g., spent solvents) or may exhibit a hazardous waste characteristic (e.g., toxicity from metals).

While the spent sampling-related equipment will be evaluated on a case-by-case basis, the general approach to be followed for spent sampling-related equipment IDW will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Containerizing the spent sampling-related equipment, typically in a satellite accumulation station.

- Characterizing the spent sampling-related equipment IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the contaminants present, and other relevant records) and best professional judgement. This characterization will be documented and maintained as part of the solid/hazardous waste determination records.

- Those spent sampling-related equipment IDW that are determined to be nonhazardous will be disposed of onsite or as municipal waste.

- Those spent sampling-related equipment IDW that are determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

6.3 Decontamination Fluids and Solids

During field investigations, decontamination fluids and solids will be generated. Typically, these will be generated at a common decon area, although there may be more than one decon

area. Typically, the decontamination IDW will include (but not limited to) washwater from vehicles/equipment, and cleaning agents. Although the vast majority of decontamination IDW is expected to be nonhazardous, this IDW may contain a listed hazardous waste (e.g., spent solvents) or may exhibit a hazardous waste characteristic (e.g., toxicity from metals).

While the decontamination IDW will be evaluated on a case-by-case basis, the general approach to be followed for decontamination IDW will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Containment of decontamination fluids (typically washwater) as generated. The washwater will be segregated from solids to the extent practicable (i.e., solids will be allowed to settle out of the washwater on the decontamination containment pad). Washwater will then be containerized to await waste determination. Solids will also be containerized in a separate container to await waste determination.

- Other decontamination solids such as cleaning utensils and PPE will also be containerized to await waste determination.

- Characterizing the decontamination IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the contaminants present, and other relevant records) and best professional judgement. This characterization will be documented and maintained as part of the solid/hazardous waste determination records.

- The decontamination solids IDW that are determined to be nonhazardous will be disposed of onsite.

- The decontamination liquids IDW that are determined to be nonhazardous will be disposed as a nonhazardous solid waste, preferably on-site.

- The decontamination IDW (either liquid or solid) that are determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

6.4 Drilling, Well Purging, and Development Water

Generally, water at the Site that is extracted from boreholes, wells or piezometers for the purpose of drilling, development, sampling, or hydraulic testing is considered non-hazardous and will be discharged to designated shallow sumps away from the boreholes or wells at the site. If the water generated is determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

7.0 PROCEDURES FOR HAZARDOUS IDW MANAGEMENT

The following procedures apply to all IDW that have been determined to be hazardous except for soil cuttings IDW that remain with the AOC unit.

7.1 Introduction

Once an IDW has been determined to be hazardous, the federal RCRA Subtitle C waste management requirements apply to that waste. The scope of this procedure covers the requirements for large quantity generators (LQG) of hazardous IDW which manage the hazardous IDW on site such that RCRA permitting is not required.

7.2 Determine Land Disposal Restrictions

The 1984 amendments to the RCRA law included a prohibition of land disposal of certain hazardous wastes without first meeting some treatment standards. For the most part, all listed and characteristic hazardous wastes must be treated according to the treatment levels and technologies outlined in 40 CFR Part 268 to reduce the toxicity and/or mobility of hazardous constituents prior to being disposed of on the land, i.e., landfilled. Therefore, a generator must

determine if the waste is a "restricted waste" under the land ban rules, and if so, off site treatment and disposal is limited. Note that these rules apply only to wastes destined for land disposal which is defined as: placement in or on the land including a landfill, surface impoundment, waste pile, injection well, land treatment facility, salt dome formation, salt bed formation, underground mine or cave, or concrete vault or bunker. Wastes which are shipped off site for disposal other than land disposal are not regulated under the land disposal restriction regulations of 40 CFR Part 268.

Generators of hazardous wastes must determine if the waste is restricted from land disposal under 40 CFR Part 268. The following reporting and recordkeeping requirements apply.

- If a generator determines that he is managing a restricted waste and the waste does not meet the applicable treatment standards, with each shipment of waste, the generator must notify the treatment or storage facility in writing of the appropriate treatment standards;
- If the generator determines that he is managing a restricted waste and the waste can be disposed without further treatment, with each shipment of waste, the generator must submit to the treatment, storage or disposal facility a notice and certification stating that the waste meets the applicable treatment standards;
- If the generator determines that he is managing a waste subject to an exemption from a prohibition on the type of land disposal method utilized for the waste, with each shipment of waste, the generator must submit to the receiving facility a notice stating that the waste is not prohibited from land disposal;
- If the generator is managing prohibited waste in tanks, containers, or containment buildings regulated under 40 CFR 262.34, and is treating such waste in such tanks, containers, or containment buildings to meet applicable treatment standards, the generator must develop a waste analysis plan which describes the procedures the generator will carry out to comply with the treatment standards; and

- If the generator determines whether the waste is restricted based solely on his knowledge of the waste, all supporting data used to make this determination must be retained on-site in the generator's files.

The generator must retain on-site a copy of all notices, certifications, demonstrations, waste analysis data, and other documentation produced pursuant to these requirements for at least three years from the date the waste was last shipped from the site. It should also be noted that it is prohibited to dilute a hazardous waste in order to circumvent the land disposal prohibitions (40 CFR 268.3). Once a waste is determined to be a "restricted waste", an appropriate Treatment, Storage, and Disposal Facility (TSDF) can be selected to properly treat and dispose of the waste.

7.3 On-Site Accumulation

As discussed in Section 5.0 above for each IDW generated, a large quantity generator (LQG) must make the appropriate hazardous waste determination per 40 CFR Part 262.11. If the IDW is determined to be hazardous, then the IDW will typically be stored on-site prior to shipment off-site for disposal. The following requirements apply to all hazardous IDW being stored on-site prior to shipment.

7.3.1 EPA Identification Number (40 CFR Part 262.12)

Any facility which is a LQG of hazardous wastes must not treat, store, dispose, transport or offer for transportation any hazardous waste without first obtaining a EPA identification number from EPA (or the authorized state). Hazardous wastes cannot be offered to transporters or to treatment, storage or disposal facilities that have not received a EPA identification number.

7.3.2 On-Site Hazardous Waste Accumulation (Storage) (40 CFR 262.34(d))

Two types of accumulation areas for hazardous waste are permissible for a LQG without RCRA interim status or a Part B permit. These are the "90-day storage area" and the "satellite accumulation station" (SAS). The SAS requirements are discussed below. With regards to a

"90-day storage area", a LQG may store hazardous wastes on-site for up to 90 days or less in a storage area, provided that the following conditions are met:

- If the waste is placed in containers, the requirements of 40 CFR Part 265 Subpart I (container requirements) are met. See below for container requirements;
- If the waste is placed in tanks, the requirements of 40 CFR 265 Subpart J (tank requirements) are met. See below for the tank requirements.
- At closure, the generator closes the storage area per the requirements of 40 CFR 265.111 and 40 CFR 265.114;
- The date which the hazardous waste is placed in the storage area is clearly marked on the container, and the container is clearly marked as "Hazardous Waste";
- The facility complies with 40 CFR Part 265 Subpart C, Preparedness and Prevention (See Section 6.3.3 below);
- The facility complies with 40 CFR Part 265 Subpart D, Contingency Plan and Emergency Procedures (See Section 6.3.4);
- The facility complies with 40 CFR Part 265.16 training requirements (See Section 6.6 below);
- Any hazardous wastes which are stored longer than 90 days must first be granted an extension by EPA (or authorized state).

90-Day Storage Area Container Requirements (40 CFR Part 265 Subpart I)

Hazardous waste stored in containers must meet the following requirements:

- Containers must be in good condition, free of leaks;
- Hazardous wastes must be compatible with container (or liner) material;
- Containers must always be kept closed except to add or remove wastes;

- Containers must be handled in a manner to avoid ruptures;
- The storage area must be inspected at least weekly to check for container deterioration; and
- Incompatible wastes must be stored separately with separate secondary containment.

Incompatible wastes are wastes that are unsuitable for co-mingling because the co-mingling could result in any of the following:

- Extreme heat or pressure generation;
- Fire;
- Explosion or violent reaction;
- Formation of substances that have the potential to react violently;
- Formation of toxic dusts, mists, fumes, gases, or other chemicals; and/or
- Volatization of ignitable or toxic chemicals due to heat generation.

90-Day Storage Area Tank Requirements (40 CFR Subpart J)

LQGs that accumulate or store hazardous wastes in tanks or tank systems must meet the following requirements:

- For tanks existing prior to July 14, 1986, an assessment of tank must be performed and certified by an independent, qualified, licensed engineer. The written certification must be kept on file at the facility (40 CFR 265.191);
- New tank systems (those built after July 14, 1986) must meet tank technical standards and have been certified by an independent, qualified, licensed engineer. The written certification must be kept on file at the facility (40 CFR 265.192);

- New tank systems must have adequate secondary containment and leak detection systems. Existing tanks must be upgraded to meet these standards by the time the tank is 15 years of age (40 CFR 265.193);
- Tanks must be operated to prevent system failure, overflow and spills. Tanks must be operated with sufficient freeboard to prevent overtopping (40 CFR 265.194);
- Inspect the tanks at least once each operating day for the following:
    - Discharge control equipment;
    - Monitoring equipment and controls;
    - Tank level; and
    - Evidence of leaks or spills. (40 CFR 265.195)
- Inspect the tanks at least weekly for corrosion, erosion or leaks;
    - The tank must meet the closure and post-closure care provisions of 40 CFR 265.197; and
    - Store incompatible wastes separately (40 CFR 265.199).

Satellite Accumulation Station (SAS) Requirements (40 CFR 262.34(c))

A SAS is a container placed at or near the point of waste generation for the purpose of collecting the waste as it is being generated. For example, a container may be placed in the quality control laboratory for collection of hazardous wastes generated in the laboratory. This SAS may collect up to 55 gallons of hazardous waste or 1 quart of acute hazardous waste. The SAS does not need to meet the requirements of a storage area, provided the following conditions are met:

- The amount of hazardous waste accumulated at the SAS does not exceed 55 gallons (or 1 quart of acute hazardous waste);

- The SAS is located at or near the point of generation where the waste is initially accumulated and is under the control of the operator of the process generating the waste;
- The container used is in good condition, is compatible with the wastes being accumulated, and is kept closed except to add or remove wastes;
- The container is marked with the words "Hazardous Waste" or other words to identify the contents; and
- Once the 55-gallon limit is reached, the date is marked on the container and the container is moved from the SAS within three days to a proper location. For example, the wastes must either be moved to the storage area or be picked up by a waste transporter and moved off-site.

7.3.3 Preparedness and Prevention (40 CFR Part 265 Subpart C)

The following preparedness and prevention steps must be taken concerning the hazardous waste storage area:

- The storage area must be operated and maintained to minimize the possibility of fire, explosions or releases of hazardous waste;
- The facility must have appropriate communication systems, fire-fighting equipment, spill control equipment and decontamination equipment;
- All emergency response systems and equipment must be tested monthly with documentation and maintained to assure proper operation;
- Persons handling hazardous wastes must have immediate access to alarms and/or communication systems;
- The storage area shall have adequate aisle space for emergency response activities; and

- The facility must attempt to make arrangements with the local police, fire departments, emergency response teams, and local hospitals to assure readiness for potential emergencies associated with the storage area.

7.3.4 Contingency Plan and Emergency Procedures (40 CFR Subpart D)

A LQG that accumulates or stores hazardous waste on site in a 90-day storage area must develop and keep current a contingency plan for the facility. The purpose of the contingency plan is to provide an organized plan of action and delegation of responsibilities and authority to specific facility personnel to respond to emergency situations that may require both the facility and/or outside resources. The contingency plan is designed to minimize hazards to humans or the environment from fires, explosion or any unplanned sudden or non-sudden release of hazardous waste/hazardous waste constituent to air, soil or surface water in compliance with the requirements of 40 CFR 265 Subpart D. A Contingency Plan will be maintained on the site if hazardous IDW are accumulated on-site.

The key components of the contingency plan include the following (40 CFR 265.52):

- A description of the emergency response organization, including designation of the Emergency Coordinator and alternates;
- Response procedures;
- Emergency notification;
- Arrangements with local authorities;
- List of names, addresses and phone numbers of designated emergency personnel and alternates;
- List of emergency response communication equipment and locations;
- Evacuation procedures, routes and alternates; and
- Procedures for amending the plan.

Copies of the plan must be sent to (40 CFR 265.53):

- The Project Manager;
- Lincoln County Sheriff's department;
- Libby fire department; and
- Other agencies as deemed appropriate.

The emergency coordinator (EC) is the key person facilitating emergency preparedness and response. The EC or designated alternate shall be on-site or on-call at all times. The EC and alternates must be trained and thoroughly familiar with the contingency plan, emergency response activities and operation of the facility. The EC must know the locations and characteristics of all waste generated, location of all records within the facility and the facility layout. The EC must have the authority to commit the resources needed to carry out the spill response plan. Any person or department who first discovers any spill of a hazardous waste/material is responsible for notifying the spill response/emergency response coordinator. The EC for the Libby Mine Site field investigations will be the EHS Officer with the Field Team Leader and the Project Manager as alternates.

The contingency plan should be reviewed and immediately amended when:

- Changes in applicable regulations occur;
- The plan fails in an emergency;
- Changes are made to emergency procedures;
- Changes occur in emergency personnel list; or
- Changes occur in emergency equipment list.

7.4 Pre-Transportation Requirements

Prior to transporting hazardous wastes or offering hazardous wastes for transportation off-site, the generator must comply with the following:

- Package the hazardous wastes in DOT-approved containers per 49 CFR Parts 173, 178 and 179. DOT-approved containers (such as drums) are usually marked as being DOT-approved);
- Label the hazardous wastes according to DOT labeling requirements per 49 CFR Part 172;
- Mark each container (of 110 gallons or less) used in transportation with the following:

  HAZARDOUS WASTE - Federal Law Prohibits Improper Disposal. If found, contact the nearest police or public safety authority or the EPA.

  - Generator's Name and Address
  - Manifest Document Number
- Ensure that the initial transporter placards the transport vehicle with the appropriate placard in accordance with 49 CFR Part 172 Subpart F.

7.5 Manifesting Off-Site Shipments of Hazardous IDW

Any generator which transports or offers for transportation hazardous waste for off-site treatment, storage or disposal must prepare a manifest according to manifest instructions for each shipment of similar hazardous wastes. The manifest must be carefully filled out with each shipment. Take care to follow the instructions and use the terms as listed in the instructions. A generator must designate on the manifest one facility (designated facility) which is permitted to handle the waste described on the manifest (40 CFR 262.20).

The generator must determine if the state to which the wastes are destined (consignment state) requires use of its own manifest. If so, then the consignment state's manifest must be used. If the consignment state does not require use of its manifest, and the state in which the waste

shipment originates (generator state) does, then the manifest from the generator state must be used. If both states have manifests, use the consignment state manifest, making sure that there are sufficient copies to meet the generator state distribution requirements. If neither state requires use of its manifest, then any uniform hazardous waste manifest may be used (40 CFR 262.21).

The manifest must contain at least enough copies such that the generator gets two copies, the transporter gets one copy and the designated facility gets one copy. Some states require additional copies to be sent to the state. At the time of shipment, the generator must keep one copy (the generator copy) of the completed, signed manifest and give the remaining copies to the transporter. Each copy must have the signature of the generator and the transporter at the time of shipment. The original manifest shall be returned to the generator once the shipment reaches the designated facility and the manifest is signed by the designated facility (40 CFR 262.21).

If the original, signed manifest is not received by the generator within a certain number of days, action by the generator is required. These requirements are discussed in the following sections:

- If, after 35 days from the date of shipment, the original manifest copy is not yet received by the LQG, the LQG must contact the transporter and/or the designated disposal facility to determine the status of the hazardous waste (40 CFR 262.42(a)(1)).
- If after 45 days from the date of shipment, the original manifest copy is not yet received by the LQG, the LQG must submit an exception report to the U.S. EPA (or authorized state). The exception report must include a copy of the manifest along with an explanation of efforts to locate the hazardous wastes and the result of these efforts (40 CFR 262.42(a)(2)).

7.6 Personnel Training

Any person, and their immediate supervisor(s), involved in waste management at a LQG facility which stores hazardous waste in a 90-day storage area must undergo initial and annual training

for hazardous waste management (40 CFR 262.34(a)(4) and 40 CFR 265.16). Facility personnel are required to successfully complete a program of classroom instruction or on-the-job training that teaches them to perform hazardous waste management duties relevant to their jobs. The program must be directed by a person trained in hazardous waste management procedures.

The training must be designed to enable personnel to effectively respond to emergencies by becoming familiar with emergency procedures, emergency equipment and emergency systems, including the following;

- Procedures for using, inspecting, repairing and replacing facility emergency and monitoring equipment;
- Communications or alarm systems;
- Response to fires or explosions; and
- Off-site communication.

Employee training is to be held at regular intervals. Emergency planning information, e.g., the Contingency Plan, also should be provided to state and local emergency response agencies at regular intervals (40 CFR 265.37 and 265.53). Employees required to receive the training cannot work unsupervised until they have completed the training requirements (either classroom or on-the-job training). In addition, facility personnel must take part in an annual review of the initial training.

The following records must be maintained at the facility for employees affected by this training:

- Job title for each position and name of employee filling each job;
- Job descriptions for each position related to hazardous waste management;
- Written description of type and amount of initial and continuing training that will be given to each person filling the various job positions; and

- Documentation that necessary training has been given and completed by each affected personnel.

Training records are required to be kept on current personnel until closure of the facility. For former employees, training records must be kept for at least three years from the date the employee last worked at the facility and may be transferred if the employee stays within the same company (40 CFR 265.16(e).

7.7 Reporting and Recordkeeping

The following reports are required of a LQG:

- Manifest exception reports as discussed in Section 6.5 above.
- A LQG must submit a Biennial Report to the EPA (or authorized state) every even numbered year by March 1, e.g., March 1, 2008 for the 2007 reporting year. The Biennial Report is to be submitted on EPA form 8700-13A.

The following records are required to be kept for a minimum of three years by the LQG:

- The signed original manifests;
- Biennial reports;
- Exception reports;
- All records pertaining to hazardous waste determinations; and
- Land disposal determination records, notification and certification records.

8.0 QUALITY ASSURANCE AND QUALITY CONTROL

All IDW data must be documented in the field logbooks, field forms, manifests, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will

check and verify that IDW documentation has been completed per this procedure and other procedures referenced herein.

9.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

EPA, 1991. Management of Investigation-Derived Wastes During Site Inspections, EPA May 1991, EPA/540/G-91/009

NOTE: This SOP has been prepared for use at the Libby Asbestos Superfund Site. The applicability of this SOP at other sites should be evaluated by the site team with regard to site-specific goals and objectives.

Date: December 6, 2007

SOP No. ISSI-LIBBY-01 (Rev. 10)

Title: SOIL SAMPLE PREPARATION

SYNOPSIS: A standardized method for preparation of soil samples for asbestos analysis at the Libby Asbestos Superfund Site is described.

Original Author: William Brattin

Syracuse Research Corporation[1]

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8:		12/11/07
Syracuse Research Corp.	WS Brattin	12/10/07

[1] This SOP was originally prepared by ISSI Consulting Group. ISSI is no longer in existence, and finalization of the SOP was performed by Syracuse Research Corporation (SRC).

REVISION LOG

Revision Number	Revision Date	Reason for Revision
1	1/7/00	Incorporation of sieving to the sample preparation.
2	7/12/00	Revision in sieve size, other minor edits.
3	5/7/02	Incorporate minor edits
4	8/1/02	Modify sieving procedure, add grinding step
5	3/6/03	Incorporate modifications to the procedure and documentation requirements
6	3/24/03	Incorporate modifications to the log-sheets to conform with electronic data storage requirements and add grinder blank requirements.
7	8/5/03	Incorporate modifications to drying and sample storage procedures
8	5/4/04	Incorporate modifications to drying batch size and recording of preparation information
9	5/14/07	Incorporate modifications so as to expand use to other Operable Units (removed references to OU4 / CSF, changed Index ID to Sample ID). Repair formatting. Remove reference to missing Figure 1. Add optional use of electronic logs. Oven temperature set to 90±10 degrees C. Lowered inventory batch size from ~120 to ~50 samples so that one inventory batch can fit in one tub. Designate drying batch as one batch per oven (~20 samples). Allow for optional use of disposable drying pans. Remove direction to NOT move grinding plates during decontamination (new BICO design allows plates to be separated for decontamination without adjusting gap). Ovens will be calibrated daily. *[Note: Revision 9 was an unsigned version that reflects changes made at the Troy Preparation Laboratory. Some of the changes in Revision 9 are retained in Revision 10, below].*
10	12/06/07	Incorporate modifications so as to expand use to other Operable Units. Designate drying batch as ~20 samples. Allow for optional use of disposable drying pans. Allow alternative methods for decontamination of plate grinder. Clarify and modify QC requirements. General editing for clarity.

1.0 PURPOSE

This Standard Operating Procedure (SOP) has been prepared by the United States Environmental Protection Agency (USEPA) Region 8 to standardize the methods used to prepare soil samples from the Libby Asbestos Superfund Site for the analysis of asbestos content. This procedure is intended for use by employees of USEPA Region 8 and by contractors and subcontractors supporting USEPA Region 8 projects and tasks for the Remedial Investigation work performed at the Libby site. Deviations from the procedures outlined in this document must be reviewed and approved by the USEPA Region 8 Remedial Project Manager or Regional Chemist.

2.0 RESPONSIBILITIES

Each laboratory that performs soil preparation activities under this SOP must have a designated Preparation Laboratory Project Leader (PL^2). The PL^2 may be an USEPA employee or contractor. The PL^2 is responsible for ensuring that all personnel in the laboratory who perform work under this SOP are familiar with the SOP, and for ensuring that all work performed satisfies the requirements of this SOP and any other relevant laboratory-specific operating procedures. It is also the responsibility of the PL^2 to communicate and document the need for any deviations from the SOP with the appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist.

All laboratory personnel preparing Libby soil samples are responsible for reading and understanding the requirement of this SOP, and for performing all applicable tasks in accordance with this SOP. Any laboratory worker who identifies any issues or encounters any difficulties in implementation of this SOP is responsible for promptly communicating the issue or difficulty to the PL^2. In addition, all laboratory personnel are responsible for reading and understanding the Health and Safety Plan (HASP) applicable to the soil preparation activities in that laboratory, and performing all tasks in accord with the requirements of that HASP.

3.0 EQUIPMENT

- General purpose laboratory oven - capable of maintaining a constant temperature of approximately 90°C.
- Analytical balance - capable of measuring in a range of 0.1 g to at least 2000 g, calibrated and accurate to the tolerance limits indicated in Attachment 2.
- Riffle splitter - with 3/4 inch chutes to split samples.

- Plate grinder - capable of accepting soil particles of approximately 1/4 inch diameter and grinding to produce particles of approximately 250 µm.

- HEPA Vacuum - A portable vacuum unit equipped with a high efficiency particulate air (HEPA) filter to remove any asbestos fibers and other soil particles from the exhaust air. Used to decontaminate equipment and maintain general laboratory cleanliness.

- Metal scoop or spoon - for transferring samples. Plastic scoops or spoons are not acceptable.

- 1/4 inch metal sieve and catch pan - for coarse sieving samples. Plastic sieves and pans are not acceptable.

- 60 mesh (250 µm) and 200 mesh (74 µm) metal sieves - for verification of the plate grinder settings. Plastic sieves are not acceptable.

- Clean quartz sand - required for preparation of grinding and drying blank samples and for decontamination of grinder.

- Clean soil - required for calibration of grinder.

- Drying pans with lids - used during the sample drying process, lids used to cover samples during transfer

- Sample containers - plastic ziplock bags (pint and gallon size).

- Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.

- Personal Protective Equipment - as specified in the applicable Health and Safety Plan for the soil preparation laboratory.

- Laboratory notebook and pen - used to record progress, any problems or observations and deviations. All information in the laboratory notebook must be recorded in pen (not pencil).

- Sample Drying Log Sheets - (Attachment 1). Used to record all sample drying information.

- Sample Preparation Log Sheets - (Attachment 1). Used to record all sample preparation information (splitting, sieving and grinding).

- Equipment Calibration and Maintenance Logs for:

  - Analytical Balance (Attachment 2)
  - Plate Grinder (Attachment 3)
  - Ventilation Hood (Attachment 4)
  - HEPA Vacuum (Attachment 5)
  - Drying Oven (Attachment 6)

  These logs are used to record all maintenance and calibration records for the listed equipment. If hard copy, all entries must be recorded in pen, and the logs must be organized and maintained in a laboratory notebook.

- Sample Labels – Self-adhesive labels for attachment to sample bags.

- Trash Bags - used to dispose of gloves, wipes and other investigation derived waste.

- Indelible Marking Pen - used to record sample information onto plastic ziplock bags and to record logbook information.

4.0 METHOD SUMMARY

Figure 1 provides an overview of the steps in the soil preparation process. Soil samples received from the field are first dried in a laboratory oven and are then split into a preparation sample and an archive sample. The preparation sample is sieved to separate coarse material (> 1/4 inch) from fine material (< 1/4 inch). The fine material is ground to a particle size of less than 250 µm, and this fine ground material is split into several aliquots. This grinding step is needed to achieve a reasonable degree of homogeneity in the sample, and to allow for preparation of slides for microscopic analysis. The coarse fraction (if any) and one aliquot of the fine ground material are then sent to an analytical laboratory for asbestos analysis by methods specified in the project-specific Sampling and Analysis Plan. At present, the fine-ground sample is generally analyzed by Phase Contrast Microscopy (Visual Area Estimation) (PLM-VE) in accord with the most recent version of SOP SRC-LIBBY-03, and the coarse material is examined by stereomicroscopy and any observable particles of asbestos are removed and weighted in accord with the most recent version of SOP SRC-LIBBY-01.

It should be noted that this preparation method, coupled with these analytical techniques, is intended to estimate the total mass fraction of asbestos that is present in a sample, without regard

to the current size distribution of the asbestos particles. That is, no distinction is drawn between asbestos that is presently in a large "lump" that is non-respirable and free asbestos fibers that are readily released to air and inhaled. Because of this, concentration values based on this approach may tend to overestimate the amount of currently releasable fibers, but do provide an estimate of the total amount of fibers that may be releasable in the future.

5.0 SOIL STORAGE

Upon receipt at the soil preparation facility, samples will be grouped into an inventory batch of 50-120 samples. Samples will be archived according to the inventory batch they are assigned to and filed by the Inventory Batch ID (box number) noted in the Sample Drying Log and Sample Preparation Log (Attachment 1).

6.0 BULK SOIL DRYING

6.1 Equipment Calibration

Samples will be weighed prior to and following drying activities. The analytical balance used for drying activities will be calibrated on days when samples are loaded into, or unloaded from, the oven. Before weighing samples, calibrate the balance using S-1 class weights and record all measurements, any required maintenance, and the balance number in the Analytical Balance Calibration and Maintenance Log (Attachment 2).

All drying activities will be performed under a negative pressure HEPA filtered hood or similar containment box. Prior to loading the oven, the ventilation hood will be calibrated to ensure that the ventilation system is operating properly. Ventilation hood calibration and any required maintenance will be documented in the Ventilation Hood Calibration and Maintenance Log (Attachment 4).

A HEPA vacuum will be used to decontaminate the oven following the removal of dried samples. Vacuum calibration will be performed daily, prior to drying activities. All system checks, required maintenance and the vacuum number will be recorded in the Vacuum Maintenance Log (Attachment 5).

Oven temperature calibration will be performed on a daily basis (during periods of operation). Oven temperature calibration and any required maintenance will be documented in the Oven Temperature Calibration and Maintenance Log (Attachment 6).

6.2 Drying Procedure

- Prior to unsealing and drying each sample, record on the Sample Drying Log the starting sample mass to the nearest 0.1 g. Include the technicians initials and the date.

- Group samples into drying batches of approximately 20 samples per batch. Assign each batch a drying batch number, and record this number on the Sample Drying Log, along with the SOP and Revision Number and the oven number used to dry the samples.

- Include one preparation blank in each drying batch. See Section 12.1 for more details regarding preparation blanks.

- Set the oven temperature to approximately 90±1°C. For every drying batch, check the oven temperature to verify that proper temperature[2] has been reached and document the start date/time and temperature in the Sample Drying Log.

- Transfer each sample to be dried from its ziplock storage bag into a clean drying pan. Each sample should be transferred to its respective drying pan under the negative pressure HEPA filtered hood. Label each drying pan with the Index ID[3] of the sample. Place each sample in the oven.

- Leave the samples in the oven for approximately 24-48 hours or until completely dry. Verify that each sample is dry by squeezing a portion of the soil with a freshly gloved thumb and forefinger to test the cohesiveness. Once it is confirmed that samples are dry, record the technician's initials, and the date and time of completion, in the Sample Drying Log.

- Turn off the oven and allow the samples to cool in the oven. Once the samples are cooled, unload each sample and transfer each sample volume to a clean ziplock bag, re-bag the sample with another clean ziplock bag and identify the dried sample with the Index ID. All samples should be transferred to ziplock bags under the negative pressure HEPA filtered hood to prevent potential exposure to fibers that might be released from the sample.

- Record the sample mass of each dried and bagged sample to the nearest 0.1 g along with the technician's initials and the date in the Sample Drying Log.

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[2] Drying temperatures in the range of 80-100°C will not compromise sample integrity, but monitoring of oven temperature to ± 1°C is needed to allow early detection of any problems with the oven temperature control.

[3] Unique sample identifiers at the Libby site are referred to as "Index ID" numbers rather than "Sample ID" numbers. However, the meaning is the same.

6.3 Decontamination

Decontaminate the inside of the hood and the inside of the drying oven by HEPA vacuuming and wet wiping all surfaces before loading a new batch for drying.

If drying pans are to be re-used, decontaminate all sample drying pans under the ventilation hood using compressed air and a HEPA vacuum to remove any residual organic material left on the pans. Wet wipe or brush off any visible material that is not removed using the vacuum.

7.0 DIVISION OF ARCHIVE AND PREPARATION SAMPLES

All dried samples are mixed and split into two portions: one portion is held in archive, and the second portion is prepared for asbestos analysis. The sections below describe the sample splitting procedure.

7.1 Equipment Calibration

Prior to any splitting, sieving, or grinding activities, calibrate the ventilation hood to ensure that the ventilation system is operating properly. Document ventilation hood calibration and any required maintenance in the Ventilation Hood Calibration and Maintenance Log.

7.2 Procedure for Sample Splitting

Splitting must be performed in the hood to prevent potential exposure to fibers that might be released from the sample. Samples will be divided using the following steps:

- Place the cooled, re-bagged samples in the hood, and knead the contents of the bag to break up any soil clumps.

- Place one collection pan on each side of the riffle splitter. Pour the sample from its plastic bag through the splitter in order to divide the sample into two equal sub-parts.

- After splitting, set aside one portion for sample preparation, as described below. If the mass of the portion for preparation is larger than about 200 grams, split the preparation sample again so that 3/4 of the original sample will be archived and 1/4 will be set aside for processing.

- Place the remaining portion(s) into a clean, ziplock bag, re-bag the sample in another clean ziplock bag, and store as an archive sample in the event additional analyses are required in the future. Identify the archive sample with the Index ID and the suffix "A" (for archive fraction). Record the technician's initials and date in the Sample Preparation

Log. Store the archive portion in the numbered inventory box noted in the Sample Preparation Log.

7.3 Preparation Duplicate Samples

One preparation duplicate sample will be prepared for every 20 field samples processed. A preparation duplicate is generated by using the riffle splitter to divide the preparation fraction into two equivalent portions ("parent" and "duplicate"). The duplicate portion is assigned an independent Index ID and both the parent sample and the duplicate sample are then processed in an identical fashion and are each submitted to the laboratory blind. For further information on preparation and processing of preparation duplicates, refer to Section 12.4.

7.4 Performance Evaluation Samples

Performance Evaluation (PE) samples are used to assess the accuracy of the analytical laboratory and to check for any potential contamination or loss of asbestos during processing. For further information on preparation and processing of PE samples, refer to Section 12.3.

7.5 Decontamination

The splitter need not be decontaminated following this step if the next use of the splitter will be the division of the fine ground fraction of the same samples into four fractions (see Section 10, below). If for any reason the next use of the splitter is division of material from a different sample, the riffle splitter must be decontaminated as follows.

- Use a HEPA vacuum and compressed air to decontaminate the splitter and brush or wipe off any visible material that is not removed by the air blast. The splitter is now ready to process the next sample.

8.0 SIEVING THE PREPARATION SAMPLE

All preparation samples are sieved prior to grinding to separate out the coarse and fine fractions. The sample sieving procedure is described in the sections below.

8.1 Equipment Calibration

All sieving activities will take place in the hood. Refer to Section 6.1 for details regarding the frequency of ventilation hood calibration.

Samples are weighed during sieving activities. The analytical balance will be calibrated daily with S-1 class weights before processing begins. All measurements, any required maintenance,

and the analytical balance number will be recorded in the Analytical Balance Calibration and Maintenance Log.

8.2 Sample Sieving Procedure

Samples will be sieved using the procedure outlined below.

- Pour the sample onto a clean 1/4 inch stainless-steel sieve with a clean pre-weighed catch pan. Shake the screen until all particles <1/4 inch in size have passed through the screen into the pan. When needed, a pestle may be used to gently break up any remaining soil clumps to ensure all particles <1/4 in size pass through the screen.

- Pour all material which does not pass through the screen (>1/4 inch) into a new, tared, sample bag. This is the Coarse Fraction.

- Weigh and record the mass of the coarse fraction to the nearest 0.1 g in the Sample Preparation Log and record the technician's initials and the date. If all of the material passes through the screen, such that there is no coarse fraction, record a mass of zero for the coarse fraction in the Sample Preparation Log.

- Double-bag the coarse sample portion and identify the sample with the Index ID and "C" suffix on the sample bag. Coarse fraction samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed.

- All material that passes through the 1/4 inch screen is the Fine Fraction. Weigh and record the mass of the fine fraction to the nearest 0.1 g in the Sample Preparation Log.

Whenever possible, immediately process the fine fraction material in accord with the approach described in Section 9.3 (below). If processing cannot occur immediately, pour the fine fraction material into a new ziplock bag and identify the fine sample material with the Index ID and the suffix "F" (for "fine fraction"). Double-bag the sample and identify the sample with the Index ID and suffix on the outside of the bag.

8.3 Decontamination

All non-disposable pans and sieves will be decontaminated between samples. Decontaminate sieves and pans (and the pestle, if used) under the ventilation hood using compressed air. Wipe or brush off any visible material that is not removed from the air blast. A HEPA vacuum may also be used to remove any residual material.

9.0 GRINDING THE FINE FRACTION

The fine fraction of each preparation sample will be ground to produce a material of about 250 µm[4]. The procedure for grinding the fine fraction is outlined below.

9.1 Equipment Calibration

All grinding activities will take place in the hood. Refer to Section 7.1 for details regarding the frequency of ventilation hood calibration.

A HEPA vacuum will be used to decontaminate the hood and processing equipment, following the preparation of each sample. Vacuum calibration will be performed daily, prior to grinding activities. All system checks, required maintenance and the vacuum number will be recorded in the Vacuum Maintenance.

A plate grinder will be used to process samples. The grinder will be calibrated daily or after any adjustments are made to the plates. To verify proper particle size (approximately 250 µm), and demonstrate that samples will not be over-processed, grind a sample of clean soil (rather than quartz sand) and sieve using stacked sieves. Clean soil will be provided by the United States Geological Survey (USGS). Unlike the coarseness of quartz sand, clean soil will more accurately approximate the typical grain size and texture of the Libby samples being processed and will reduce the chance of over-processing.

The grinder is adjusted acceptably if, after grinding of the clean soil sample, all material passes through a 60-mesh (250 µm) screen and is substantially retained by a 200-mesh (74 µm) sieve. If a significant amount of the ground clean soil sample is retained on the 60-mesh screen, or if a substantial fraction of the material passes through the 200-mesh screen, adjust the plates of the grinder until these targets are achieved. If the required particle size cannot be achieved even after plate adjustment, other grinder maintenance such as plate replacement may be required. Regardless, grinding of field samples cannot resume until the desired particle size is achieved. Document the grinder number, verification of acceptable adjustment and any observations in the Grinder Calibration and Maintenance Log.

Samples will be weighed following grinding activities. The analytical balance will be calibrated daily with S-1 class weights before processing begins. All measurements, any required maintenance, and the analytical balance number will be recorded in the Analytical Balance Calibration and Maintenance Log.

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[4] Note that the particle size is cited as "approximately 250 µm". This is due to the nature of grinding asbestos material. Some material that is longer than 250 µm may pass through the grinder if its longest side is parallel with the vertical grinder plates. The material that comes in contact more nearly perpendicular to the vertical grinder plates will be ground to <250 µm

9.2 Grinding Blanks

One grinding blank per grinder will be prepared daily, and will be associated with all samples prepared by that grinder on that day. For further information on grinding blanks refer to Section 12.2.

9.3 Grinding of Fine Field Samples

The sample portion that was sieved to < 1/4 inch will be ground to a particle size of approximately 250 µm. Set up a catch pan under the grinder to collect all the ground material. Take the fine sample set aside in Section 8.2, load the grinder hopper, and allow the fine sample to pass through the plate grinder into the catch pan. Note the technician's initials, date of grinding, and grinder number in the Sample Preparation Log.

The net recovery of fine ground material must not be less than 90% of the mass of fine material placed into the grinder. If recovery is less than 90%, soil grinding must be stopped and the grinder re-adjusted until the mass recovery of test sand and/or soil samples exceeds 90%.

9.4 Decontamination

Plate Grinder

The details of decontamination of the plate grinder and it associated containers and equipment may vary depending on the model of grinder that is being used.

If the plate grinder can be readily disassembled for cleaning without altering its grinding properties, disassemble the grinder and clean the chutes and plates with the HEPA vacuum and compressed air. Then, if needed, use wet wipes to ensure decontamination. If wet wipes are used, the plates and chutes must be thoroughly dried before reassembly. If the grinder is not easily disassembled, clean the grinder with the HEPA vacuum and several blasts of compressed air, paying special attention to areas where dust from the grinding process is known to accumulate (e.g., between the plates and areas adjacent to the catch pan clamps). Then, pass an aliquot of approximately 20 g of quartz sand through the grinder to clean out any residual soil. Discard the quartz sand and re-clean the grinder with the vacuum and another round of high pressure air blasts. After this decontamination procedure, the grinder is ready to process the next sample.

In general, all soil containers, hoppers and catch pans associated with use of the grinder should be decontaminated by using a HEPA vacuum and/or wet wipes, followed by a blast of high pressure air.

Calibration Sieves

The stacked sieves used to calibrate the plate grinder will be decontaminated using a HEPA vacuum and compressed air between calibration uses.

10.0 SPLITTING OF THE FINE GROUND SAMPLE

The fine ground soil sample should be distributed into four approximately equal subsamples using a splitter. All splitting activities will be performed in the hood. Refer to Section 7.1 for details regarding the frequency of ventilation hood calibration.

10.1 Splitting Procedure for Fine Ground Sample

The following method for splitting a soil sample was adapted from EPA 540-R-97-028 (USEPA, 1997):

- Set up one receiving pan on each side of the splitter. Load the soil from the grinder catch pan (Section 9.3) into the splitter, collecting the sample in two receiving pans.

- Tap the catch pan vigorously several times to free any remaining material. Tap the splitter to facilitate the flow of all material through the chutes into the receiving pans.

- Empty one receiving pan into the grinder catch pan and the other receiving pan into the sieve catch pan. Set the sieve catch pan aside; this portion of fine ground sample will be split again later.

- Replace the receiving pans under the splitter. Take the grinder catch pan, containing half of the fine ground sample, and re-load the contents into the splitter as detailed above. Repeat the process of dispersing the sample material by shaking the catch pan and tapping the splitter to uniformly distribute the sample. The resulting splits are the "FG1" and "FG2" portions in the Sample Preparation Log.

- Take these two portions and carefully transfer each into a clean, tared, ziplock sample bag. Re-bag one sample portion in another clean ziplock sample bag and identify this fine ground sample with the Index ID, the suffix "FG" (for "fine fraction, ground") and the fraction number 1, (ex. CS-12345-FG1 for fine ground fraction #1). Identify the bagged second portion with the Index ID, the suffix "FG" and the fraction number 2 and set aside to be re-bagged with the following fine ground portions:

- Place the two empty receiving pans from the "FG1" and "FG2" portion next to the splitter. Repeat the splitting procedure using the other fine ground portion set aside in the sieve pan and split the remaining sample material to create the "FG3" and "FG4" portions.

- Take the remaining "FG3" and "FG4" portions and carefully transfer each into a clean, tared, ziplock sample bag, identify each remaining fine ground sample with the Index ID as noted above.

- Weigh each sample portion (FG1 through FG4), and record each mass along with the technician's initials and date in the Sample Preparation Log.

Combine all of the bagged coarse and fine portions of the sample into one large clean, ziplock sample bag.

Coarse and fine ground samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed. When samples are requested for shipment, the "FG1" fraction will be sent first. If further analyses are required for the fine ground portion, the subsequent fractions will be double bagged and sent (i.e., FG-2 then FG-3, etc.). All archived fine ground portions will be filed in the appropriate inventory archive box noted in the Sample Preparation Log.

10.2 Decontamination

The splitter must be decontaminated between each sample. Use the vacuum and/or wet wipes followed by a blast of compressed air to decontaminate the splitter and brush or wipe off any visible material that is not removed by the vacuum or air blast. The splitter is now ready to process the next sample.

11.0 DOCUMENTATION

Index ID numbers are recorded in the Sample Drying Log, Sample Preparation Log and on all sample containers. Sample Drying Logs and Sample Preparation Logs will be filed or archived according to their associated dry batch and preparation batch number. If revisions to the Sample Drying Log and/or Sample Preparation Log are necessary, the appropriate parties will be notified of the changes, however, these changes will not necessitate revision to the current standard operating procedure, a modification form will be filled out to document the revisions.

As mentioned above, the following equipment calibration and maintenance logs will also be maintained:

- Daily analytical balance calibration using S-1 class weights (Attachment 2)
- Daily grinder setting verification for calibration check and/or post-adjustment verification, grinder maintenance as necessary (Attachment 3)
- Daily ventilation hood operating condition verification (i.e., inline filter checks, changes) (Attachment 4)
- HEPA vacuum maintenance and bag changes (Attachment 5)
- Weekly oven temperature calibration, oven maintenance as necessary (Attachment 6)

In addition, a laboratory notebook will be maintained by each individual or team that is preparing samples. For each day that samples are processed, the following information should be collected:

- Date
- Time
- Personnel
- Personal protective equipment (PPE)
- SOP (including revision number) and any other laboratory-specific governing plan being followed
- Descriptions of any deviations to the SOP, the reason for the deviation and/or any modification forms being followed
- Summary of laboratory activities (including number of samples prepared, and equipment calibrated and used)

12.0 QUALITY CONTROL

Quality control (QC) samples are inserted into the sample train to monitor for potential contamination introduced during the preparation process or to assess accuracy of analysis that may be affected due to preparation procedures. If samples results indicate the occurrence of contamination or inconsistent results, the PL^2 will be notified. The PL^2 will then notify the EPA Regional Project Manager and the Regional Chemist in order to review laboratory procedures and identify any changes in preparation laboratory methods and procedures that may be necessary. Any such reviews and resultant changes will be documented accordingly by the PL^2.

12.1 Preparation Blanks

A preparation blank is a sample of 200-400 grams of clean quartz sand that is treated identically to a field soil sample. That is, the preparation sample is dried in the oven along with the field soil samples, split into archive and preparation fractions using a riffle splitter, screened through a ¼ inch screen (even though there are no particles larger than ¼ inch), and ground by passing through the plate grinder. This type of sample is intended to detect contamination that may occur at any stage of the soil preparation procedure.

At least one preparation blank will be processed with each drying batch of approximately 20 field samples. Preparation blanks will be assigned a random and unique Index ID and will be submitted to the laboratory blind. The Index ID assigned to each preparation blank must be in accord with the numbering system specified in the program-specific project plan.

Detection of asbestos fibers (any type) in any preparation blanks at a level greater than Non-detect (Bin A) by PLM-VE should be taken as a sign of potential cross-contamination, and all field samples associated with the preparation batch for the preparation blank having detectable asbestos (> Bin A) will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any of the corresponding field samples. If the overall fraction of preparation blanks that contains detectable asbestos (> Bin A) exceeds 1%, a review of laboratory procedures should be undertaken to identify and address the source of the contamination.

12.2 Grinding Blanks

A grinding blank consists of 100-200 grams of clean quartz sand that is passed through the plate grinder. The purpose of this type of sample is to evaluate the effectiveness of decontamination procedures for the plate grinder.

One grinding blank per grinder will be prepared for each day that field samples are being ground. Each grinder used in the laboratory will be assigned a number and all samples processed will be associated with the grinder used for preparation. The grinder number used for each sample will be noted in the Sample Preparation Log. Grinding blanks will not be dried, split for archive, or sieved. Rather, a grinding blank will only be ground and split into four fine ground samples. The grinding blank is assigned a random and unique Index ID and is submitted to the laboratory blind. The Index ID assigned to each grinding blank must be in accord with the numbering system specified in the program-specific project plan.

Detection of asbestos fibers (any type) in any grinding blank at a level greater than Non-detect (Bin A) should be taken as a sign of potential cross-contamination, and all field samples associated with the grinding blank that reports detectable asbestos (> Bin A) will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any pf the corresponding field samples. If the overall fraction of grinding blanks that contains detectable asbestos (> Bin A) in a soil preparation facility exceeds 1%, steps should be taken to develop an improved method for grinder decontamination.

12.3 Performance Evaluation Samples

Performance Evaluation (PE) samples are samples of Libby soil that have been spiked with a known amount of Libby Amphibole (LA) asbestos. These samples were prepared by the USGS

for use at the Libby site by spiking uncontaminated soil from Libby with a known mass of LA fibers collected at the mine site, and then grinding the sample to a particle size of ≤ 250 um as described above. Several different concentration values of PE samples were prepared, ranging from < 0.1% to 2%. Each bottle contains about 100 grams of the PE material.

PE samples will be utilized in two ways.

First, the soil preparation facility will insert untreated PE samples into the analytical sample train sent to the laboratory for PLM-VE analysis. This type of PE sample is intended to evaluate the performance of the analytical laboratory (rather than the preparation facility).

Second, the soil preparation laboratory will process PE samples in the same way that field soil samples are processed, as detailed below. This type of PE sample is intended to determine if there is any loss of asbestos during sample processing. In addition, considered in conjunction with a grinding blank that is passed through the decontaminated grinder immediately following the PE sample, the PE sample will also be used to facilitate assessment of grinder decontamination procedures.

The frequency of each type of PE sample (unprocessed and processed) should be one per month for each month in which soil processing is occurring. These should be distributed approximately evenly between the different concentration values that are available for PE samples.

Each month that soil processing is occurring, the procedure to be followed for generation and submittal of PE samples is as follows:

1. Select a PE bottle for inclusion.
2. Thorough mix the contents of the PE bottle by inversion (a minimum of 10 times) and/or rolling (a minimum of 10 minutes).
3. Remove an aliquot of about 20 grams and package this for submission to the analytical laboratory without any processing. If more than one laboratory is analyzing samples, rotate the submittal of unprocessed samples so that all laboratories receive approximately equal total number of unprocessed PE samples.
4. Take the remainder of the PE bottle (about 80 grams) and carry this material through the full sequence of steps applied to each field sample, starting with oven drying. After splitting the dried sample with the riffle splitter, recombine the samples so that the full 80 grams is screened through the ¼ inch sieve and passed through the plate grinder. Thus, there is no archive split for PE samples. After grinding and splitting, this should result in four sub-samples of processed PE sample. Prepare three of these for submittal to the analytical laboratories, and hold one sample in archive.

Results of PE samples processed by the soil preparation laboratory are evaluated by comparing the reported results for LA to the nominal results. Deviations from nominal may be the result of variations either in soil processing procedures and/or in the analytical procedure. If the frequency of strongly discordant results (i.e., the results of the PE sample differ by more than one bin from the nominal result) exceeds 10%, then the source of the inconstancy should be investigated and remedied.

12.4 Preparation Duplicates

A preparation duplicate is prepared by using a riffle splitter to divide a field soil sample into two approximately equal portions, creating a parent and duplicate sample. Both samples are then processed in the same fashion. The preparation duplicate is assigned a unique Index ID, and is submitted to the laboratory blind. The Index ID assigned to each preparation duplicate must be in accord with the numbering system specified in the program-specific project plan.

One preparation duplicate sample will be processed for every 20 field samples prepared (5%). Results from duplicate samples serve to evaluate the precision of the combined sample preparation process and the laboratory analysis. Inconsistent results between parent and duplicate may be due either to variability in sample preparation, sample analysis, and/or to small scale variability in the sample that is not fully controlled by mixing and splitting. If the overall frequency of strongly discordant results (i.e., the results for the parent sample and duplicate are different by more than one bin) is greater than 10%, steps should be taken to identify and address the source of the variability in the sample preparation procedure.

13.0 DECONTAMINATION

All non-disposable equipment used during soil sample preparation must be decontaminated prior to use. Scoops, spoons, splitters, sieves and drying pans that are re-used must be decontaminated with a HEPA vacuum, compressed air, wet-wiping and/or by brushing off any residual material. If soil particles are visible on any of the equipment, repeat the decontamination procedure until the equipment is clean. To reduce the potential for human exposure in the laboratory, COMPRESSED AIR SHOULD BE USED CAREFULLY AND ONLY UNDER VENTED HOODS.

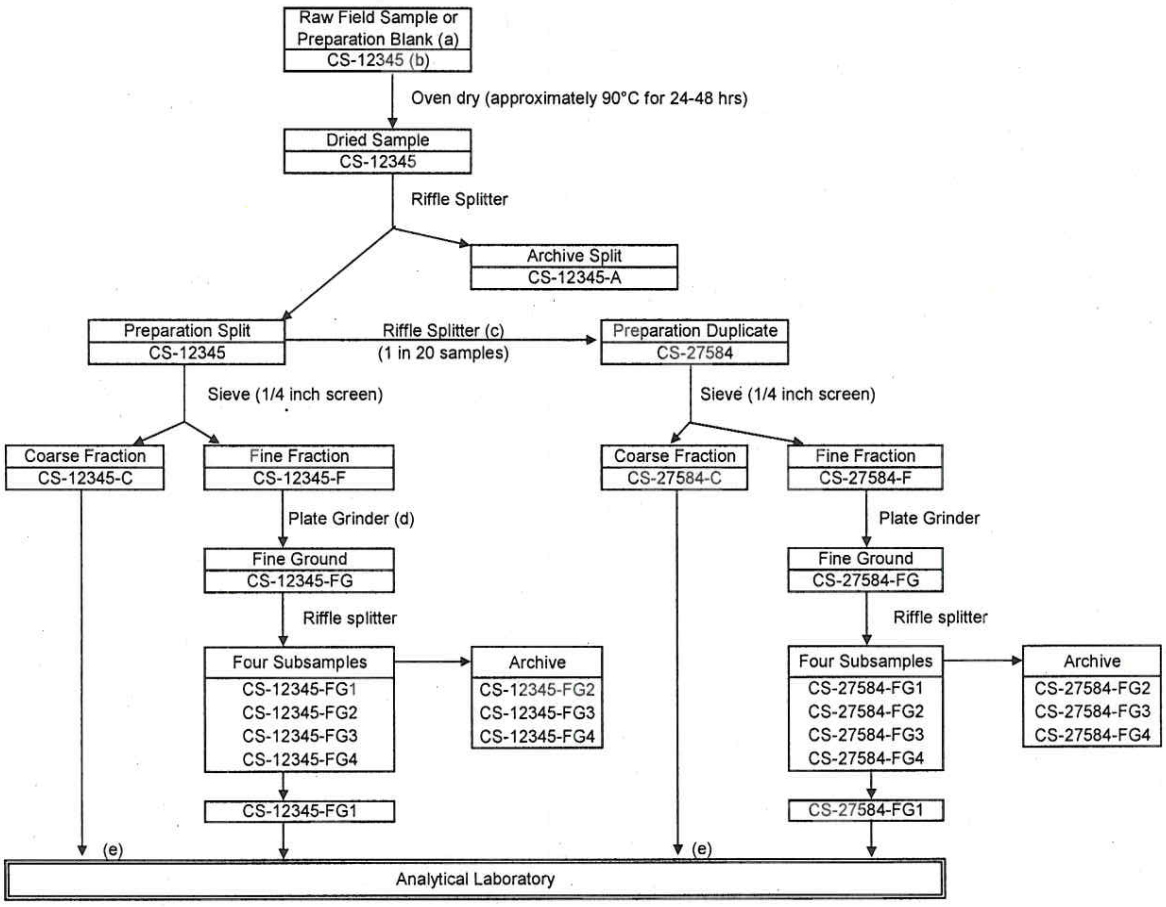
Detailed decontamination procedures for specific equipment are noted in Sections 6.3, 7.5, 8.3, 9.4, and 10.2.

14.0 REFERENCES

American Society for Testing and Materials. 1998. Standard Practice for Reducing Samples of Aggregate to Testing Size, ASTM Designation: C 702 - 98, 4 p.

USEPA. 1997. Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials. EPA 540-R-97-028.

FIGURE 1 SOIL PREPARATION FLOW DIAGRAM



NOTES:

(a) A preparation blank (200-400 grams of clean silica sand) is prepared in the same way as field samples at a rate of 5%

(b) Example Index ID (sample number) shown to illustrate naming conventions

(c) A preparation duplicate is prepared at a rate of 5%

(d) A grinding blank (100-200 grams of clean sand) is passed through the plate grinder and split into 4 sub-samples at a rate of 5%

(e) Coarse sample will be returned to EPA for archive after laboratory analysis

ATTACHMENT 1

SAMPLE DRYING AND SAMPLE PREPARATION LOG SHEETS

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Sample Drying Log Sheet

Laboratory Name: _____ Sheet No.: _____

Drying Begun: date _____ time _____

Drying Complete: date _____ time _____

Oven number: _____

Oven temp: _____ °C

	Index ID	Inventory ID No.	SOP and Rev No.	Sample mass (g)			Original Sample ID and Notes (indicate if preparation blank)	QC
				Before drying	After Drying	Initials and date		Initials and Date
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

Sample Preparation Log Sheet

Laboratory Name: _____ Sheet No.: _____

Preparation Batch: _____

	Index ID	SOP and Rev No.	Inventory ID	Drying Batch ID	Archive Sample Splitting	Duplicate Sample Splitting	Sieving			Sample Grinding		Sample Splitting					Original Sample Identification and Notes (indicate if grind blank, prep blank, or duplicate pair. For duplicate pair enter the parent ID)	QC
							Sample Mass (g)					Sample Mass (g)						
					Initials and date	Initials and date	Coarse Fraction > 1/4"	Fine Fraction < 1/4"	Initials and Date	Initials and Date	Grinder #	FG1	FG2	FG3	FG4	Initials and Date		Initials and Date
1																		
2																		
3																		
4																		
5																		
6																		
7																		
8																		
9																		
10																		
11																		
12																		
13																		
14																		
15																		
16																		
17																		
18																		
19																		
20																		

The following preparation steps require Technician Initials and Date to document activity: Sample Drying, Archive Sample Splitting, Preparation Duplicate Splitting, Sieving, Homogenization, Sample Splitting

ATTACHMENT 2

ANALYTICAL BALANCE CALIBRATION AND MAINTAINANCE LOG SHEET

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Preparation Laboratory = _____

Balance # = _____

Measurement Number		S - 1 Class Weight Measurements				Measurement within range? Yes or No	If "No" Recalibrate	Technician Initials	QC check initials
	Calibration Weights	0.1 g	1 g	10 g	100 g				
	Tolerance Limit Range	0.05 - 0.15 g	0.90 - 1.10 g	9.75 - 10.25 g	99.00 - 101.00 g				
	Date								
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									

The analytical balance calibration will be verified daily.
All tolerance limits are standard tolerance limits for Class S-1 weights.
After 20 measurements, the tolerance range will be evaluated for reasonableness.
Weights falling outside the range require that the balance be recalibrated using all S-class weights

Sheet No.: Balance - _____

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ATTACHMENT 3

GRINDER CALIBRATION AND MAINTAINANCE LOG SHEET

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Preparation Laboratory = _____

Grinder # = _____

Date	Activity Type (D, A, M)	Sieve Test: 60 mesh acceptable Y or N	Sieve Test: 200 Mesh acceptable Y or N	Notes (Include description of action/maintenance)	Technician Initials	QC Check Initials

D = daily calibration
A = Adjustment Calibration Verification
M = Maintenance Only
For the 60 mesh sieve test to be acceptable all of the clean soil must pass through the sieve.
For the 200 mesh sieve test to be acceptable a substantial portion of the ground soil must be retained on the sieve.
Failure of either sieve test requires adjustment of the plates followed by adjustment verification prior to grinding samples.

Sheet No.: Grinder - _____

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ATTACHMENT 4

VENTILATION HOOD CALIBRATION AND MAINTAINANCE LOG SHEET

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Preparation Laboratory = ________________________________

Ventilation Hood # = ___________________

Date	In-line HEPA indicators acceptable?	Notes (Include description of action/maintenance)	Technician Initials	QC Check Initials

Ventilation system operating conditions should be verified daily
In-line system should indicate the velocity/negative flow is 100 ft/min. If not, the problems with the system must be corrected prior to processing samples. Note actions or maintenance taken.

Sheet No.: Hood - _______________

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ATTACHMENT 5

HEPA VACUUM CALIBRATION AND MAINTAINANCE LOG SHEET

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Preparation Laboratory = ______________________________

Vacuum # = __________________

Date	System Check Acceptable? Yes or No	HEPA filter clean? Yes or No	Bag fill level acceptable? Yes or No	Notes (Include description of action/maintenance performed)	Technician Initials	QC Check Initials

Physically check the vacuum suction and note audible change in motor daily. If significant reduction in the vacuum suction or motor "strain" is audibly noted then the system check is unacceptable. If unacceptable, perform and document the HEPA filter and bag maintenance checks above and perform maintenance.

Sheet No.: Vacuum - ______________

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ATTACHMENT 6

OVEN CALIBRATION AND MAINTAINANCE LOG SHEET

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Preparation Laboratory = _____

Oven # = _____

Date	Certified Temperature Reading (°C)	Thermometer ID	System Check Acceptable? (90+/-1 °C) Yes or No	Notes (Include description of action/maintenance performed)	Technician Initials	QC Check Initials

Sheet No.:Oven - _____

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LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE

APPROVED FOR USE IN LIBBY SUPERFUND SITE ONLY

Date: April 21, 2004 SOP No. SRC-LIBBY-01 (Rev. 2)

Title: QUALITATIVE ESTIMATION OF ASBESTOS IN COARSE SOIL BY VISUAL EXAMINATION USING STEREOMICROSCOPY AND POLARIZED LIGHT MICROSCOPY

Author Sally M. L. Gibson Syracuse Research Corporation

SYNOPSIS: A standardized method is described for the examination of the coarse fraction (>1/4") of soil samples using stereomicroscopy and polarized light microscopy (PLM) to identify, segregate, and estimate the mass percent of asbestos in the sample matrix.

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8		4/26/04
Syracuse Research Corp.		4/26/04

Revision	Date	Reason for Revision
0	11/12/02	--
1	5/20/03	Provided clarification on dealing with very small particles.
2	4/21/04	Included statements on limitations of intended use

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized screening method for the visual examination of the coarse fraction of previously sieved soil samples for evidence of asbestos mineral content using stereomicroscopy with confirmation of asbestos content by polarized light microscopy (PLM). This SOP incorporates salient components of EPA Test Method 600/R-93/116 *Method for Determination of Asbestos in Bulk Building Materials* and National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM*, Issue 2.

This procedure will be used by employees of contractors/subcontractors supporting USEPA Region 8 projects and tasks for the Libby, Montana, site. Deviations from the procedure outlined in this document must be approved by the USEPA Region 8 Remedial Project Manager or Regional Chemist prior to initiation of sample analysis.

2.0 PREREQUISITE TRAINING

Visual examination will be performed according to this SOP by a laboratory accredited by the National Voluntary Laboratory Accreditation Program (NVLAP) and by analysts proficient either by education or experience in asbestos mineral identification by stereomicroscopy and PLM. Analyst familiarity with the procedural applications prescribed in EPA Test Method 600/R-93/116 and NIOSH Method 9002 is required.

Training as described in the Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4, (CSS SQAPP [CDM 2002]) will be provided to laboratory personnel or laboratories with less than one year of project-specific experience by "mentors" from either Reservoir Environmental Services, Inc. or EMSL.

3.0 RESPONSIBILITIES

The CDM Laboratory Coordinator (LC) is responsible for overseeing the activities of the CDM Soil Preparation Laboratory and subcontracted laboratories performing sample analysis for the Libby, Montana, project. The LC is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the CSS SQAPP. It

is the responsibility of the LC to communicate with the project personnel and subcontracted laboratory regarding specific analysis objectives and anticipated situations that require any deviation from the CSS SQAPP SOPs. In addition, it is the responsibility of the LC to communicate the need for any deviations from this SOP with the CDM Project Manager, USEPA Region 8 personnel (Remedial Project Manager or Regional Chemist.)

Subcontracted laboratory analysts performing the visual examination are responsible for adhering to the applicable tasks outlined in this SOP and substantiating components of the reference procedures (EPA 1993; NIOSH 1994) with the modifications contained herein.

4. 0 EQUIPMENT

- Analytical balance - accurate to 0.01 g, range of 0.01 g to 1000 g (for weighing total sample)

- Analytical balance - accurate to 1 mg (for weighing asbestos)

- Traceable standards - major asbestos types

- Microscope - binocular stereomicroscope, 5-60X approximate magnification

- Microscope - polarized light, binocular or monocular with a cross hair reticle (or functional equivalent) and magnification of at least 8X
  - 10X, 20X, and 40X objectives
  - 360 degree rotatable stage
  - substage condenser with iris diaphragm
  - polarizer and analyzer which can be placed at 90 degrees to one another and calibrated relative to the cross-line reticle in the ocular
  - port for wave plates and compensators
  - wave retardation plate (Red I Compensator) with ~550 nanometer retardation and known slow and fast vibration directions

- Light Sources - incandescent or fluorescent

- Tweezers, dissecting needles, scalpels, probes, razor knives, etc. - standard sample manipulation instruments/tools

- Microscope slides and cover slips

- Refractive index liquids

- Pre-tared glassine paper, glass plates, weigh boats, petri dishes, watchglasses, etc. - laboratory sample containers

- HEPA-filtered or Class 1 biohazard hood negative pressure

- Three-ring binder book- binders will contain Microscopic Examination Logbook Sheets (Attachment 1)

5.0 METHOD

Soils from the Libby, Montana site will be dried, sieved, and prepared according to the most recent revision of SOP ISSI-LIBBY-01, Soil Sample Preparation. The coarse fraction of the soil sample is defined as that portion of the sample which does not pass through a 1/4" sieve. The coarse fraction will be weighed, placed in a zip-top plastic bag, and labeled as described in Camp, Dresser, and McKee (CDM) SOP 1-3 (with project-specific modifications). The samples will be packaged and shipped by the soil preparation laboratory as described in CDM SOP 2-1 (with project-specific modifications) and transferred to the laboratory via chain-of-custody procedures described in CDM SOP 1-2 (with project-specific modifications).

The following sections describe the stereomicroscopic and PLM examination. Materials tentatively characterized as asbestos by stereomicroscopy will be isolated and subjected to confirmation by PLM. The mass % of Libby amphibole asbestos, other amphibole asbestos, and chrysotile asbestos in the coarse soil fraction will be calculated from the mass of each asbestos type positively identified by PLM and the original sample weight. Figure 1 provides an overview of the process.

5.1 Stereomicroscopic Examination

The laboratory will receive the coarse fraction soil samples from the CDM Soil Preparation Laboratory. The entire sample will be weighed and placed in an appropriate container. The weight of each coarse sample will be recorded, along with the sample identification, on the Microscope Examination Logbook Sheet. The sample will be subject to stereomicroscopic examination and particle segregation as depicted Figure 1. The stereomicroscopic examination to identify and segregate asbestos includes:

- using multiple fields of view over the entire sample
- probing the sample by turning pieces over and breaking clumps where possible
- manipulating the sample using appropriate instruments/tools
- observing homogeneity, texture, friability, color and extent of any observed asbestos in the sample(s)

NOTE: Although the coarse fraction is prepared by sieving with a 1/4" screen, particles smaller than 1/4" may be present in the fraction due to adherence between coarse and fine particles. This may even include some very fine asbestos fibers. Because of the technical difficulty, the analyst should not attempt to physically segregate and weigh particles smaller than about 2-3 mm (1/10 inch). A particle this size is expected to have a mass of about 10-20 mg, which is less than 0.1% of a sample whose total mass is 25 grams. If no particles larger than 2-3 mm are present, this should be noted in the data sheet for each category of asbestos using the following code system:

- ND = No asbestos observed
- Tr = Trace levels of asbestos observed but not quantified

The weight fraction for any asbestos type marked “ND” or “Tr” in a given sample is not calculated and is left blank.

As the sample is examined, the analyst will continue segregation of the sample until the entire coarse soil fraction has been characterized as either “non-asbestos” or “tentatively identified asbestos.” The tentatively identified asbestos particles will be examined by PLM, as described below. The stereomicroscopist will initial and date the Microscopy Examination Logbook Sheet.

5.2 PLM

The coarse material tentatively identified as asbestos by stereomicroscopic examination will be subject to confirmation using PLM, as described in SOP SRC-LIBBY-03 (Revision 0) ("Analysis of Asbestos Fibers in Soil by Polarized Light Microscopy"). The PLM examination will be used to confirm that the particles tentatively classified as asbestos are actually asbestos, and will be assign each particles to one of three categories:

LA = Libby amphibole
OA = Other amphibole
C = Chrysotile

If OA is observed, the type of OA observed should be noted in the data sheet using the following code system:

- AMOS = Amosite
- ANTH = Anthophyllite
- CROC = Crocidolite
- UNK = Unknown

The total weight of each type of positively identified asbestos (LA, OA, C) will be determined and recorded on the Microscopic Examination Logbook Sheet, along with the analyst's initials and the date of the examination.

6.0 QUALITY ASSURANCE

Laboratories performing the examination must be accredited by NVLAP. "Calibration" should be verifiable for each microscopist in terms of project-specific training and the successful analysis of materials of known asbestos content (NVLAP test samples, in-house standards) similar to those anticipated to be observed in Libby, Montana soils. Additionally, references such as photographs of the asbestos minerals illustrating distinguishing properties should be available benchside during characterization.

Quality control samples as described in ISSI-LIBBY-01 (i.e., preparation duplicates) will not submitted for the coarse materials samples. The entire coarse fraction will be subject to examination.

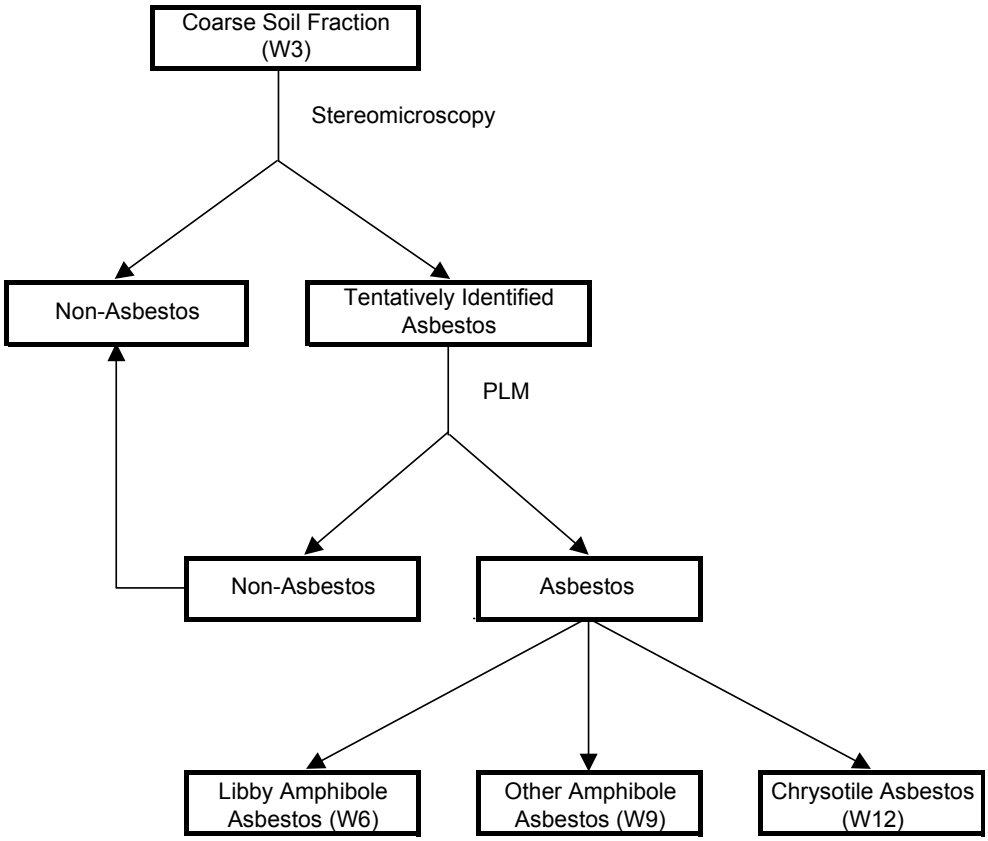
7.0 REFERENCES

CDM 2002. *Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4.* 3282-116-PP-SAMP-14187. Camp, Dresser and McKee Denver, Colorado. April.

NIOSH 1994. National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM*, Issue 2.

USEPA 1993. *Method for Determination of Asbestos in Bulk Building Materials*. 600/R-93/116.

Figure 1. Overview of Sample Examination Process



W3 = Original coarse soil fraction mass (g)
W6 = If present in measurable quantities, mass (mg) of Libby amphibole
W9 = If present in measurable quantities, mass (mg) of other amphibole
W12 = If present in measurable quantities, mass (mg) of chrysotile

Codes used in the illustration (e.g., W3) correspond to Data Log Sheet

ATTACHMENT 1

MICROSCOPIC EXAMINATION LOGBOOK SHEET

SRC-LIBBY-01 Data sheet and EDD.xls
(Check with Volpe or SRC to determine the latest version number)

Example hard copy of data entry sheet shown on next page (for illustration purposes only).

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Data Log Sheet v6 for SOP SRC-LIBBY-01

Stereomicroscopic and Gravimetric Analysis of Coarse Soil

Lab Name:

SOP Version:

Lab Job No.

Calculated automatically in the "Electronic Data Entry" form. Do not enter data here.

EPA Index ID	Index Suffix	Lab Job-Sample No.	Status (1 = Analyzed, 2 = Missing, 3 = Contam, 4 = Cancelled)	QA Type (Not QA)	Total Sample Weight (g)			Analysis Details		Mass of Asbestos Particles (mg)																		Comments (see Notes below)
										Libby Amphibole (LA) Asbestos				Other Amphibole (OA) Asbestos					Chrysotile (C) Asbestos									
					Tare Weight (g) Empty Container	Mass of Sample + Container (g)	Mass of Sample (g)	Analyst Initials	Analysis Date	LA Qual* (ND, Tr)	Tare Weight - Container (mg)	Mass of LA + Container (mg)	Mass (mg) LA	OA Qual* (ND, Tr)	OA Type** (AMOS, ANTH, CROC, UNK)	Tare Weight - Container (mg)	Mass of OA + Container (mg)	Mass (mg) OA	C Qual* (ND, Tr)	Tare Weight - Container (mg)	Mass of C + Container (mg)	Mass (mg) C	% LA	% OA	% C	

Notes:

*Qualifier codes: ND = No asbestos observed.
Tr = Trace levels observed but not quantified.

**OA Type codes: AMOS = Amosite
ANTH = Anthophyllite
CROC = Crocidolite
UNK = Unknown

Comment Codes (user-defined):

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Date: July 21, 2005 SOP No. SRC-LIBBY-03 (Revision 2)

Title: **ANALYSIS OF ASBESTOS FIBERS IN SOIL BY POLARIZED LIGHT MICROSCOPY**

Author: William Brattin

SYNOPSIS: A semi-quantitative method for identifying and quantifying asbestos fibers in soil using polarized light microscopy (PLM) is provided. This method is based on NIOSH Method 9002, EPA Method 600/R-93/116, and CARB Method 435, with project-specific modifications intended specifically for use at the Libby Superfund Site.

APPROVALS:

TEAM MEMBER	**SIGNATURE/TITLE**	**DATE**
USEPA Region 8		
Syracuse Research Corp.		

Revision	Date	Principal Changes
0	03/03/03	--
1	12/11/03	Clarify binning assignment of samples at 0.2%
2	07/21/05	Provided more detail on refractive index for oils

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standard approach for semi-quantitative analysis of asbestos in samples of soil or other soil-like materials using polarized light microscopy (PLM). This SOP is specifically intended for application at the Libby Superfund site.

2.0 SCOPE AND APPLICATION

This method is intended mainly for analysis of asbestos in soil or other similar soil-like media. This method is appropriate for the analysis of all types of asbestos fibers, including both chrysotile and amphiboles, including those that are characteristic of the Libby site.

3.0 RESPONSIBILITIES

It is the responsibility of the laboratory supervisor to ensure that all analyses and quality assurance procedures are performed in accord with this SOP, and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation or analysis. The laboratory supervisor should also communicate with project managers at EPA or their oversight contractors any situations where a change from the SOP may be useful, and must receive approval from EPA for any deviation or modification from the SOP before proceeding with sample preparation and analysis.

4.0 METHOD DESCRIPTION

The soil sample to be evaluated for asbestos content by PLM is examined under stereomicroscopy and under PLM (3-5 slides per sample) to estimate the amount of asbestos present. Quantification of the amount of asbestos present may be done either using a visual estimation approach or by a point counting approach, as specified in the Chain of Custody request. In either case, the concentration of Libby amphibole asbestos in the sample is estimated in terms of mass fraction (i.e., percent asbestos by weight) based on the use of project-specific reference materials (calibration standards).

5.0 DETAILED METHOD

5.1 Basic Methods

All qualitative and quantitative analyses are to be performed in general accordance with the methods and techniques specified in NIOSH 9002, EPA 600/R-93/116, and CARB Method 435. Project-specific modification, clarifications, and requirements are provided below.

5.2 Visual Estimation Approach

5.2.1 Classification of Asbestos Mineral Type

Based on fiber attributes (morphology, refractive index, color, birefringence, etc.), asbestos in the sample is classified into one of three categories:

Code	Description	Notes
LA	Libby Amphibole	Refractive index values for LA span the standard values for tremolite/actinolite (EPA 1993), but may include values for other similar amphiboles (e.g., winchite, richterite) characteristic of the mine at Libby. Based on analysis of 4 different samples from the mine (Wylie and Verkouteren 2000; USGS, unpublished data; Verkouteren, personal communication), **observed refractive indices of Libby amphiboles** range from about 1.629-1.640γ and 1.614-1.623α, with a birefringence of about 0.017. The full range of refractive indices of samples from the mine may be somewhat greater. **Based on these data, when confirming the identity of LA, the target refractive index (RI) for oil is about 1.620-1.630. See Attachment 2 for details on preparing oils of a specified RI.**
OA	Other amphibole	Includes amphibole forms (e.g., amosite, crocidolite, anthophyllite) that are not thought to occur in significant amount at the mine in Libby
C	Chrysotile	

5.2.2 Estimation of LA Mass Percent

The visual area estimation is a semi-quantitative approach that requires the microscopist to estimate the area fraction of the total material present in a field of view that consists of asbestos material. Because this estimation may be difficult, especially at low concentration values, and because the desired output is an estimate of mass fraction (rather than area fraction), all visual estimates of Libby amphibole content will be performed using a set of site-specific reference materials (calibration standards) as a frame of reference. These reference material will contain either 0.2 % or 1.0% Libby amphibole by weight[1], and have been prepared for analysis using the same approach as for field samples. Using the two reference concentrations (0.2% and 1.0%) as a visual guide, the microscopist will evaluate the field sample and report the results as follows:

PLM Laboratory Report			Description
Qual	Conc (wt.%)	Bin	
ND		A	Asbestos was not observed in the field sample
Tr		B1	Asbestos was observed in the field sample at a level that appeared to be lower than the 0.2% reference material
<	1	B2	Asbestos was observed in the field sample at a level that appeared to be approximately equal to or greater than the 0.2% reference material but was less than the 1% reference material.
	1, 2, 3, etc	C	Asbestos was observed in the field sample at a level that appeared to equal or exceed the 1% standard. In this case, the mass percent is estimated quantitatively.

[1] The nominal mass fraction of the reference materials (calibration standards) is based on the gravimetric fraction of the material that is soil and the amount that is spiking material, adjusted for the fraction of the spiking material that is LA. For example, if the spiking material were estimated to contain 85% LA by mass, then the 1.0% calibration standard would contain 1.18 grams of spiking material (1.00 grams of LA) per 100 grams of calibration standard. Because the estimate of LA content of the spiking material is approximate, the true concentration of a calibration material may not be precisely equal to the nominal value.

"ND" (not detected) in the Qualifier column is used for all samples in which asbestos is not observed under stereomicroscopy and is also not detected in five (5) different PLM slides prepared using representative sub-samples of the test material. These samples are assigned to **Bin A**.

"Tr" (trace) in the Qualifier column is used for all samples in which asbestos is observed either under stereomicroscopy or in at least one out of 3-5 PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be less than the 0.2 % reference material. These samples are assigned to **Bin B1**.

"<" (less than) in the Qualifier column and 1 in the Concentration column is used for all samples in which asbestos is observed either under stereomicroscopy or in PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be equal to or greater than the 0.2 % reference material but less than the 1% reference material. These samples are assigned to **Bin B2**.

A numeric value (1, 2, 3, etc) in the Concentration column without an entry in the Qualifier column is used for all samples in which asbestos is observed either under stereomicroscopy or in PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be similar to or greater than the 1 % reference material. These samples are assigned to **Bin C**.

Note that because these reference materials are based on Libby amphibole, they are not appropriate for estimating the mass percent of other types of asbestos (chrysotile, other types of asbestos). Therefore, if any asbestos types besides Libby amphibole are observed, the reported values for those samples should be in units of area percent.

5.3 Point Counting Approach

5.3.1 Counting Procedure

Any analysis in which evaluation by point counting is requested will be performed in general accordance with the descriptions provided in EPA/600/R-93/116 and CARB Method 435. The

total number of particles to be counted (generally 400 or 1000) will be specified in the Chain of Custody request.

Take eight sub-samples of the soil sample and mount each separately with the appropriate refractive index liquid. The preparations should not be heavily loaded. Each sample should be uniformly dispersed to avoid overlapping particles and allow 25-50% empty area within the fields of view.

An ocular reticule (point array) or cross-hair is used to visually superimpose points on the microscope field of view. Count 1/8 of the total points required on each of the 8 slides (e.g., 50 non-empty points per slide for a 400 point count and 125 non-empty points per slide for a 1000 point count). For each non-empty point counted, assign the particle that is present at the point into one of four bins:

- Not asbestos
- Libby asbestos (LA)
- Other asbestos (OA)
- Chrysotile asbestos (C)

In order for a particle to be counted as asbestos, the aspect ratio must be $\geq$ 3:1.

After the required total number of non-empty points have been counted, record the total number of points in the LA, OA and C bins on the point counting data sheet.

5.3.2 Estimation of Mass Percent

Like visual estimation, the output of the point counting approach is an estimate of area fraction, not mass fraction. For this site, point-count estimates of area fraction for Libby amphibole particles will be converted into estimates of mass fraction using a standard curve approach.

The standard curve will be prepared using a series of site-specific reference materials (calibration standards) containing 0%, 0.2%, 0.5%, 1%, or 2% Libby amphibole. The area fraction of each reference material will be estimated by the point counting approach in quadruplicate. The standard curve will be prepared by plotting the mean area fraction

determined by point counting versus the mass percent in the reference material. The mass fraction of a field sample will be determined by measuring the area fraction of the field sample and locating the mass fraction that corresponds to that area fraction on the standard curve.

Because the standard curve is based on Libby amphibole, it is not appropriate to utilize this standard curve for other types of asbestos. Therefore, if any asbestos types besides Libby amphibole are observed, the reported values for those samples should be in units of area percent.

6.0 APPARATUS AND MATERIALS

Polarized light microscope, with lens and filters
Stereomicroscope (approximately 10-45x)
Petri dish for stereomicroscopic sample examination
Spatula and forceps
Glass slides and cover slips
Refractive Index (RI) oils
Reference Materials (Calibration Standards)
- Soil containing 0.2% LA by mass
- Soil containing 0.5% LA by mass
- Soil containing 1.0% LA by mass
- Soil containing 2.0% LA by mass

Laboratory log book
Data recording sheet (Attachment 1)
Liquid calibration conversion table (Attachment 2)

7.0 QUALITY ASSURANCE/QUALITY CONTROL

7.1 Precision and Accuracy

PLM by visual estimation and point counting are both semi-quantitative methods. For the purposes of this project, the accuracy and precision of the method are evaluated by measuring the frequency with which samples are assigned to the correct "bins". Data on precision and accuracy of bin assignment will be collected in the future and used to establish performance criteria for this project.

7.2 Method Proficiency

At present, sufficient data are not available to establish a quantitative procedure for method proficiency demonstration. As results become available, a procedure will be established and applied, based on the analysis of a set of blind Performance Evaluation materials and assessing the frequency of correct bin assignments. If the assignments reported by a laboratory are within acceptance criteria bounds (see Section 7.1), then that laboratory will be deemed proficient. If not, remedial actions must be taken to address the errors before work may begin by that laboratory.

8.0 RECORDS

8.1 PLM Data Forms

Analysts will record analytical results using the electronic data sheets developed for the Libby project, as presented in Attachment 1. Note that there are two different electronic forms; one is for use in visual area estimation, and the other is for use in point counting. Once completed and checked, these spreadsheets are submitted to EPA for upload into the database. The laboratory should retain all original records for use in resolving any questions until otherwise instructed by EPA.

8.2 Instrument Maintenance Logbook

An individual instrument maintenance logbook should be kept for each piece of equipment in use at the laboratory. All maintenance activities must be recorded in the appropriate logbook.

8.3 Data Storage and Archival

Electronic Data. Each day of data acquisition, all electronic files will be saved onto two separate media. For example, the data may be saved onto a computer hard drive, but must also be backed up onto a type of portable media such as CD-ROM, floppy disc, or tape. Portable media will be maintained in a single location with limited access.

Hardcopy Data. All data sheets and micrographs must be stored in a secured location with limited access (e.g., locking file cabinet) when not in use.

Copies (hardcopy and electronic) of the raw analytical data will be submitted to USEPA for archival.

9.0 REFERENCES

CARB 435. California Environmental Protection Agency Air Resources Board, Method 435, Determination of Asbestos Content in Serpentine Aggregate. June 6, 1991.

EPA. 1993. Method for the Determination of Asbestos in Bulk Building Materials. United States Environmental Protection Agency, Office of Research and Development. EPA/600/R-93/116. July 1993.

EPA. 2003. Technical Memo 8. Procedure for Combining Mass Fraction Estimates for Coarse and Fine Fractions of Soil. Prepared by US EPA Region 8 with technical assistance from Syracuse Research Corporation.

NIOSH. 1994. Asbestos (Bulk) by PLM. NIOSH Manual of Analytical Methods, Fourth Edition. National Institute of Occupational Safety and Health. August 15, 1994.

Wylie AG and Verkouteren JR. 2000. Amphibole Asbestos from Libby, Montana: Aspects of Nomenclature. American Mineralogist 85:1540-1542.

ATTACHMENT 1

PLM DATA RECORDING SHEETS

PLM (VE and PC) Data Sheet and ED.xls
(Check with Volpe or SRC to determine the latest version number)

ATTACHMENT 2

RI Liquid Calibration Conversion Tables
Prepared by Dr. Shu-Chun Su, Hercules, Inc.

See attached Excel spreadsheet entitled
"Create_RI_Liquid_Calibration_Conversion_Tables.xls"

Date: May 27, 2008 OU3 FISH-LIBBY(Rev. 0)

Title: FISH SAMPLING

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	_____	_____
SOP Author	_____	_____

Revision Number	Date	Reason for Revision
0	05/27/2008	--
1		

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) describes the protocols to be followed when fish are collected for biological surveys, chemical analysis of tissues and/or histopathological examination. The procedures presented herein apply to sediment sampling from surface waters, wetlands, ponds, drainage structures, etc.

This document focuses on methods and equipment that are readily available and typically applied in collecting fish samples. It is not intended to provide an all-inclusive discussion of fish collection methods. Specific sampling problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be clearly described in the project-specific sampling plan and approved by the Project Manager and the Quality Manager.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in sediment sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Environmental Sample: A solid sample collected for chemical or geotechnical analysis. These samples are used to support remedial investigation, feasibility studies, treatability studies, remediation design and performance assessment, waste characterization, etc.

Net Seine: Seine nets are constructed of mesh panels hung from a float line with a weighted lead line attached to the lower edge.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects site-specific sampling methods, sample locations, and constituents to be analyzed with input from other key project staff.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Biologist: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL, field biologist, or engineer in the implementation of tasks. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

5.0 METHOD SUMMARY

This section describes sediment sampling from the bottom of a surface water drainage course or pond. The collected samples will be placed in appropriate sample containers, as designated by the FSP or Quality Assurance Project Plan (QAPP), for transfer to a laboratory for the analyses identified in the FSP. Details of sample collection will be described on the attached sediment sampling form.

6.0 EQUIPMENT

The selection of sampling equipment listed above depends on the site conditions and sample type

required. In addition, the following equipment is needed to collect fish samples:

GENERAL

Field notebook, indelible marker
Global Positioning System (GPS) unit
Marking stakes
Digital Camera
Compass
100 m measuring tapes
Detergent solution (0.1-0.3 % Alconox)
Distilled water
Latex gloves
Ziploc bags
Paper Towel
Chain of custody and sample labels
Coolers
Sample bottles
Plastic sheeting

Equipment needed for fish collection is listed below, by procedure.

SEINING

Seine
Buckets
Carpet needle and string
Waders
Wader belts

ELECTROFISHING

Backpack electroshocker
Battery
Waders
Buckets
Wader belts
Fiberglass handled dip nets

FISH PROCESSING

Data Sheets	Measuring board
Balance or scale	Field guides or keys
Coin envelopes	Knife
Forceps	Saw
Probe	Pliers
Ziploc® bags	Aluminum foil

Large scissors	Small scissors
Dissecting microscope	Glass scintillation vials with lids
Glass jars with lids	Preservative
Scalpel	Fillet knives
Knife sharpener	Dissecting trays

7.0 SAMPLE COLLECTION

Fish samples will be collected using either seining and/or electroshock collection methods as specified in the following subsections. Attachment A provides a Field Sample Data Sheet (FSDS) for recording field information on each fish sample. [Note: in some cases, an alternative FSDS may be specified and provided in the project specific SAP]. Note any special circumstances or conditions about the sampling location. Obtain and record the GPS coordinates of the sampling location on the FSDS form.

7.1 Seining

Seine nets are constructed of mesh panels hung from a float line with a weighted lead line attached to the lower edge. Seines are selective sampling gear, and will not capture all sizes of fish. The size of fish you want to sample will determine the mesh size of the seine. Mesh size should be small relative to the target fish. Too large a mesh size will allow fish to escape through the net, however mesh sizes too small will be difficult to pull through the water. Seines are most effective in water no deeper than two-thirds the height of the net.

The net should have a pole at each end which is at least equal to the height of the net. Poles should be held at a 45o angle away from the direction of movement when pulling the seine.

For sampling a stream, the seine should be long enough to reach from bank to bank. Unless stream flow is very low, the seine is pulled against the current. Care should be taken to run the poles holding the seine directly along the bank, and under it if the bank is undercut. The leadline must remain in contact with the bottom to prevent fish from escaping under the net, and the float line must stay on or above the water surface. Several fish species (e.g. largemouth bass) will attempt to jump over the top of the seine when confined, so the float line should be above water when these are the target species.

After a collection is made, both seiners should walk onshore and pull the leadline up immediately. If there is no convenient place to beach the seine, the leadline can be lifted above water by both collectors at the same time. After the net is out of the water, captured fish should immediately be transferred to water-filled containers.

In a lake, a seine may be pulled parallel to the shore or from offshore toward the shore. Alternatively, one end of the seine can be planted on the bank, and the other end can be pulled out, around, then back in to the bank.

Mesh size and length of a seine will determine size of fish which can be caught, and may affect how efficiently the seine can be pulled. Mesh sizes too small will be difficult to pull, especially if there is much debris in the water. High current velocity in a stream will also decrease seining effectiveness.

To prevent fish from escaping under or over the net, it is imperative that the leadline be kept in contact with the bottom, and the float line must stay on or above the water surface. Streams or lakes with rocky bottoms or debris that snags the leadline will be difficult to seine effectively. Having a third person follow the seine and free it from snags helps prevent losing fish when the seine gets caught.

Seines can be torn as they are pulled through the water, leaving holes through which fish can escape. The seine should be inspected frequently, and repaired as necessary.

7.2 Electrofishing

Use of electricity to capture fish is one of the least selective of all active fish capture methods. This method involves creating an electrical field in the water by passing a current between two submersed electrodes. There are two types of electrical current. DC always flows in one direction because the negative and positive ends (electrodes) of the circuit do not change. Direct current will induce galvanotaxis (forced swimming with orientation) and fish will move toward the anode. With ac, the anode (the positive electrode) and the cathode (the negative electrode) switch positions, so the current flows alternatingly in both directions. Fish exposed to ac will be stunned and lose equilibrium, and can be easily netted.

Electrofishing can be done using a backpack-mounted electroshocker unit, a shore-based unit, or from a boat. Backpack shockers are best for small streams. A minimum of three people are needed, one to run the shocker and two dip netters. The crew should wade upstream, with the dip netters beside or behind the electrode handler. All stunned fish, regardless of size or species, should be collected. The sampling area should be fished slowly and methodically, especially areas with in-stream cover. Captured fish should be placed in water-filled buckets. Nets can be set at the upper and lower ends of a stream section to prevent movement of fish out of the sample area.

Shore-based electrofishing is similar to backpack shocking, except that the power source stays onshore. Shore-based fishing is more dangerous, as voltages of shore-based units are higher than backpack units. The crew is also separated from the power source, and may not have safety switches. A buddy system should always be used during a shore-based electrofishing operation.

When electrofishing from a boat, the electrodes are suspended from a boom off the front of the boat. The boat should be driven slowly through shallow areas or along weed beds, and one or two people should stand near the bow and dip net stunned fish.

Research objectives, habitat characteristics and availability of the power source will influence the choice of current to be used. DC should be used when it is important not to damage or kill fish, and is very effective in turbid water or in thick weeds or brush. AC generators are generally less bulky, and are effective in clear unobstructed water. AC is more harmful to fish than DC, and may cause hemorrhaging, rupture swim bladders or fracture vertebrae.

Both direct and alternating currents can be modified to produce various current shapes that have different effects on fish. Pulsed DC will sustain forced swimming with less damage to fish. In addition, pulsed DC requires less voltage than ac and a smaller electrical source can be used. Pulsed ac will have the same effect as unmodified AC, but is not as potentially harmful to fish.

Water conductivity will affect the efficiency of electrofishing. In water where the conductivity ranges between 100 and 500 micromhos/cm, electrofishing will be most effective. At high conductivities, water is less resistive than fish and the current will flow around them. Electrofishing is not used in salt water habitats. Low conductivity water is more resistant than

fish, and the electrical field is limited to the immediate area of the electrode.

Environmental factors which can affect electrofishing include water conductivity, temperature, season, and time of day. Electrofishing success is poor in water with very high or low conductivity. Electrofishing is most effective in shallow habitats. If water temperatures are high, some fish species may move into deeper water where temperature is lower and oxygen is higher. During spawning season, some species may be captured in shallow areas that would normally be found in deeper areas. Electrofishing at night catches more species, larger individuals, and more fish than similar effort during the day.

Because batteries and generators used for electrofishing provide more than enough current to electrocute a person, it is vital that safety rules be observed. All members of an electrofishing crew should understand the system and the risks involved. One person should be in charge of the operation, and this person should control the power source. Shut down the power source before any repairs or equipment changes are made. Electrofishing should never be done alone, and the crew and power source should stay close together.

8.0 SAMPLE PREPARATION AND ANALYSES

Fish can be collected for multiple purposes. Fish may be collected to identify if contaminants in aquatic habitats accumulate in fish tissue, cause histopathological damage, or affect fish condition or growth. Impacts on aquatic community structure can also be assessed. The specific procedures used to process fish will depend on the project objectives. The following sections describe analyses of biological parameters, tissue contaminant analyses, and histopathology.

8.1 Measuring and Recording Biological Parameters

Data the may be recorded on collected fish in the field include length, weight, species, and information on parasites or other abnormalities. When possible, sex and stage of maturity should also be noted. Data are recorded on a data sheet (Table 1) specific to the sampling location. All members of the processing staff should be trained in techniques used to make length and weight measurements as well as external examination. Inconsistencies in the way these measurements

are taken can lead to errors.

Length. Fish length is measured using a measuring board on which the anterior end of a fish is placed against a stop at the beginning of a measuring scale. The fish should be measured with mouth closed, and the body positioned on its right side with the head to the measurer's left. Any one of three measurements can be taken: total, fork or standard length (Figure 1). Total length is the greatest length of a fish from its anterior most extremity (usually the mouth) to the end of the tail fin. For fish with a forked tail, the two lobes should be pressed together, and the length of the longest lobe should be taken. Fork length is measured from the anterior end of the fish to the tip of the middle rays of the tail. Standard length is the length of a fish from the anterior end of the fish to the tip of the middle rays of the tail. Standard length is the length of a fish from the anterior end to where the base of the median tail fin rays join the caudal peduncle. This spot can be located by bending the tail sharply. A crease should form where the tail fin rays end. Total length or fork length measurements are used most often. Determination of standard length is very difficult on some species. Factors which contribute to length measurement errors are musclular tension in live fish, eroded fins, shrinkage of fish due to preservation, and failure to consistently squeeze the tail to get maximum total length.

Weight. Spring balances or electronic digital scales are generally used to weigh individual fish. Fish can be weighed by themselves, or by placing them in a container of water. Taking the weight in water reduces error due to fish movement, but may not be practicable for large fish. Large numbers of fish can be weighed in bulk if individual weights are not needed (e.g., for population studies).

Because most fish maintain near-neutral buoyancy in water, their specific gravity is close to 1.0 and body volume is proportional to weight. Therefore, the amount of water displaced in a container can also be used to determine weight. When taking weights, an attempt should be made to have fish at a standard degree of wetness. Variation in stomach contents or amount of water swallowed at capture will also affect fish weights. Other sources of error include movement of the scale due to fish movements, wind or boat motion.

Species. Study objectives will dictate what level of identification is needed for a fish. Fish collected should be identified to species level. Local authorities should be consulted before field work begins to determine whether regional taxonomic references exist.

Sex and Age of Maturity. Information on the sex and stage of maturity should be recorded for each fish where possible. If fish are collected during spawning season, some fish can be sexed based on breeding colors. Mature fish may release eggs or milt when they are handled.

Gross Pathology. All fish collected will be examined for any external signs of abnormalities or parasites. The following may be included in the gross external examination: examination of body surface and denote as normal or abnormal. Abnormalities may include excess mucus or irregular color.

8.2 Gross Fish Necropsy

A gross necropsy may be completed in the field or laboratory in fish sacrificed for further external and/or internal examination and/or the collection of tissue samples (described in Section 8.3). These procedures are taken from the Bozeman Fish Health Center SOP No. MISC 236.1 AADAP dated September 21, 2006 for gross fish necropsy and the internal examination of fish. The general steps for fish necropsy include:

A. Fish should be euthanized with a chemical anesthetic, in an ice-water bath, or by spinal severance before being examined.

B. Depending upon the size of the fish, examination of external and internal features should be done with the unaided eye or with a dissecting microscope, whichever is appropriate.

C. Examine the body surface of the fish, and denote as normal or abnormal. Abnormalities may include excess mucus or irregular color.

D. Document presence of lesions on body surface, whether lesions were open or closed, and location of any such lesions.

E. Examine gills by lifting opercula, and document whether normal or abnormal.

Abnormalities may include pale or hemorrhagic gill tissue.

F. Place the fish on its right side on cutting board or pan. The fish should be opened using three cuts to expose the internal organs.

G. Observe position of organs, looking for presence of flu Observe position of organs, looking for presence of fluid in the cavity and for free parasites. If fluid is present, the color and turbidity of fluid should be recorded.

H. Peritoneum and mesenteries: These are normally transparent, smooth, glistening and most. Check for hemorrhaging, parasites, thickening, turbidity. Document and findings.

I. Digestive system:

- Cut the hind gut just anterior to the anus. Disconnect the liver from the cavity walls. Remove the esophagus and remove the digestive organs.
- Separate the liver from the stomach and observe it color, surface, and whether margins are thickened. Cut the liver and observe the color, consistency and presence of nodules and parasites.
- If desired, and fish are big enough, open the stomach and intestine with scissors. Record whether food is present. Wash the wall of the digestive tract and observe for edema, hemorrhaging and inflammation.

J. Check the size, color and margins of the spleen.

K. Air bladder: If desired, remove the air bladder and check for content (liquid present?), appearance, and for presence of parasites.

L. Kidneys: Examine the anterior and posterior kidneys for color, size (swelling), and presence of nodules or parasites.

G. Other tissues that may be examined include eyes (for exopthalmia), the body cavity (for colored fluid), gall bladder, adipose, and musculature.

8.3 Tissue Sample Preparation

In some cases, tissues may be collected from the sampled fish for either analyses of contaminant levels and/or histopathological examination. Analyses may be performed on whole body or specific organs and or tissues. A FSDS should be completed for each tissue sample processed. Sampling location, tag number, date, species, and data on the specimen metrics described below should be recorded. A gross necropsy should be performed as described in Section 8.2 for each

fish prior to collection of tissue samples. Procedures for tissue sample collection and preparation are described below.

Whole Body. Analyses of contaminant levels in whole body may be performed. Based on the objectives of the study, the stomach contents of the fish may be removed (using dissection technique) prior to analysis. Alternately, fish may be held in aerated chambers for 24 hours to depurate stomach contents. This will allow for a determination of the concentration of contaminants accumulated in the tissue versus contaminants entrained in the gut.

Filleting. To assess risk to humans from fish consumption, fish are typically filleted and only muscle tissue sent to the laboratory for analysis. To fillet a fish, an initial cut should be made from the dorsal fin to the pelvic fin, just behind the opercular flap. Run the tip of the knife along the dorsal side of the fish, from the initial cut to the caudal fin. Continue making successively deeper cuts, running the knife blade as close to the neural spines and ribs as possible. After the fillet is obtained, remove the skin. Place the skin side of the fillet down on the dissecting tray, hold on to the tail portion of the fillet, and run the knife between the skin and the muscle tissue. Turn the fish over and repeat the process to obtain the other fillet.

Dissecting. Fish are dissected if specific tissues and/or organs are being collected for hisopathology or for contaminant residue analysis. Begin the dissection by laying the fish on its right side and making an incision from just above the vent to the top of the rib cage. Cut along the rib cage, forward through the pectoral girdle. Make a shallow incision to avoid damage to internal organs. Pull the flap downward to open the body cavity. Note any gross abnormalities or parasites observed in the body cavity. Also record, if possible, sex and stage of maturity.

Liver, gill and kidney tissues are the fish tissues collected most often for histopathology or residue analysis. The liver should be located near the anterior end of the stomach. It is connected to the gut by the gall bladder and bile duct. The liver should be removed and weighed to the nearest 0.001 g. A hepatosomatic index, liver weight expressed as a percentage of body weight, can be used as an indicator of fish condition. For histopathology, two tissue sections should be obtained from the distal end of the medial lobe. The sections should be cut 1.0 centimeter (cm) towards the center of the lobe, and 0.5 cm thick. Cut the section using a scalpel,

and handle carefully to avoid crushing the tissue. Place the tissue sections in a Whirl-Pak bag or screw top plastic container (USGS 1999) filled with 4 percent buffered paraformaldehyde.

The gills are located beneath the opercular flap. Pull back or remove the operculum to expose the gills. Carefully remove a section of gill tissue, taking care not to crush it. Place the tissue sections in a glass scintillation vial filled with 4 percent buffered paraformaldehyde. Buffered paraformaldehyde can be purchased through commercial chemical supply companies.

The kidney is located along the backbone above the gas bladder. Kidney tissue is difficult to remove from fish because it adheres to the body wall and is soft. Thin slices can be taken through the vertebral column which include the kidney. These tissue sections should be preserved with the liver and gill tissue sections. Again, for proper preservation, the specimen volume should be no more than 50 percent of the total volume occupied by specimen and preservative.

Tissue samples for residue analyses may be collected either in the field or in the laboratory (from whole fish shipped from the field). Tissue samples for histopathology should be collected and preserved in the field prior to shipment to the laboratory. Extreme temperatures can alter tissue characteristics, making tissues unsuitable for analysis. Exposure of dead specimens to extreme cold can cause tissue to freeze, making histopathological analysis difficult. Extreme heat can cause rapid decomposition of tissue. An effort should be made to keep fish alive until they are processed. Samples should be labeled and shipped following procedures outlined in the Sample Documentation and Sample Packaging and Shipping SOPs.

8.3 Histopathology

Samples collected and preserved for histopathology should be transported to a laboratory qualified and experienced in performing hispathology examination of tissues. The histopathology laboratory will be responsible for further fixation and preparation of samples for histopathological examination.

9.0 QUALITY ASSURANCE AND QUALITY CONTROL

All sampling data must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and the other procedures referenced herein. All equipment must be operated according to the manufacturer's specifications, including calibration and maintenance. If possible, species identifications will be confirmed by a regional biologist familiar with the site aquatic fauna.

10.0 DECONTAMINATION AND HEALTH AND SAFETY

All equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

All field crew members will conduct sampling in accordance with the appropriate level of health and safety training required by their parent organization.

Any time fish are collected, water and boat safety precautions must be taken. Wading can be hazardous in swift currents or if the bottom is uneven or algae-covered. Falls can be avoided by moving slowly, taking short steps, and wading sideways to the current. Guidelines for boating safety should be followed for all activities which require transportation by boat.

Safety procedures which should be observed while electrofishing include use of the buddy system, clear communication between the sampling team, and all samplers in waterproof gloves and waders which do not leak. The electrofishing equipment should be equipped with 'dead man' automatic shut-off switches, and one person should control the power source. At least one member of an electrofishing team must be certified in CPR.

11. 0 REFERENCES

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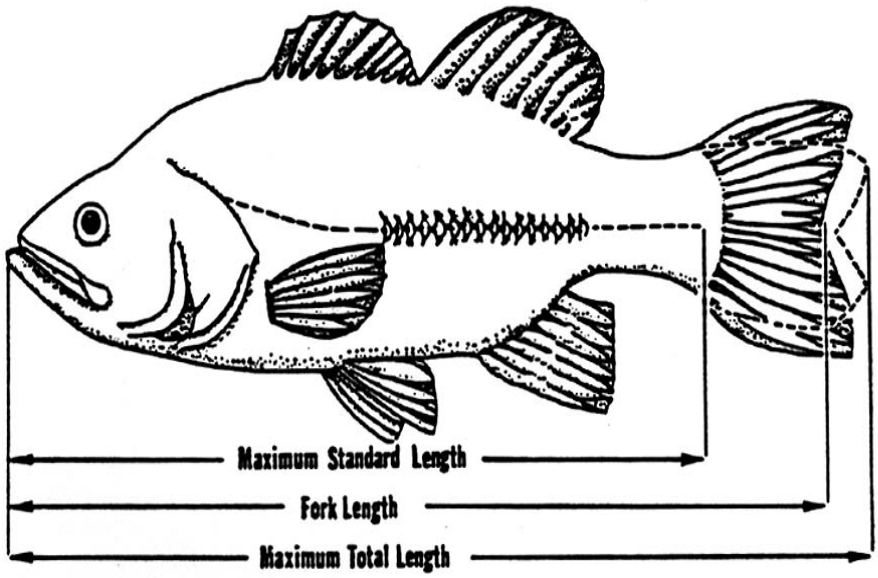
FIGURE 1. Measurements of Fish Length - Standard, Fork, and Total
(From Anderson and Gutreuter 1983)



Date: May 27, 2008 SOP#BMI-LIBBY-OU3 (Rev. 0)

Title: BENTHIC MACROINVERTEBRATE SAMPLING AND PROCESSING

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	_____	_____
SOP Author	_____	_____

Revision Number	Date	Reason for Revision
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TECHNICAL STANDARD OPERATING PROCEDURE
BENTHIC MACROINVERTEBRATE SAMPLING & PROCESSING

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Physical Characterization/Water Quality Field Data Sheet

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ATTACHMENT 2 Sample Log-in Sheet

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TECHNICAL STANDARD OPERATING PROCEDURE
BENTHIC MACROINVERTEBRATE SAMPLING & PROCESSING

SYNOPSIS: A standardized method for collecting benthic macroinvertebrates for ecological assessment. This procedure is based on USEPA Rapid Bioassessment Protocol III. Protocols for sample collection and handling are provided.

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for sampling the benthic population at hazardous waste sites. This protocol summarizes the USEPA Rapid Bioassessment Protocol III (RBP III) for benthic macroinvertebrates. RBP III utilizes the systematic field collection and analysis of major benthic taxa, and can detect subtle degrees of impairment at potentially contaminated sites. Discrimination of four levels of impairment should be possible with this assessment. This SOP may be used by employees of USEPA Region 8, or contractors and subcontractors supporting USEPA Region 8 projects and tasks. Deviations from the procedures outlined in this document must be approved by the USEPA Region 8 Remedial Project Manager, Regional Toxicologist or On-Scene Coordinator prior to initiation of the sampling activity.

2.0 RESPONSIBILITIES

The Field Project Leader (FPL) may be an USEPA employee or contractor who is responsible for overseeing the benthic macroinvertebrate sampling activities. The FPL is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the FPL to communicate with the Field Personnel regarding specific collection objectives and anticipated situations that require any deviation from the Project Plan. It is also the responsibility of the FPL to communicate the need for any deviations from the Project Plan with the appropriate USEPA Region 8 personnel (Remedial Project Manager, Regional Toxicologist or On-Scene Coordinator).

Field personnel performing benthic macroinvertebrate sampling are responsible for adhering to the applicable tasks outlined in this procedure while collecting samples.

3.0 EQUIPMENT

- D-frame dip net - 0.3 m^2 "D"-shaped net (500um nytex screen) where the net attaches to a long pole. Net is cone-shaped for capture of organisms.

- Kick-net - 1 m^2 net (500um nytex screen) attached to 2 poles, which functions in a similar manner to a fish kick seine.

- Ponar/Ekman Dredge - for collection of sediment-dwelling invertebrates in non-wadable waterbodies.

- Surber sampler - 12 by 12 inch square sampling grid with attached 500 um nytex mesh net.

- Elutriator Bucket or 500 um Mesh Sieve Bucket - used to remove invertebrates from sediment.

- Collection containers - wide-mouth bottles (500 to 1,000 ml capacity).

- Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex; should be disposable and powderless.

- Field notebook - a bound book used to record progress of sampling effort and record any problems and field observations during sampling.

- Three-ring binder book- to store necessary forms used to record and track samples collected at the site. Binders will contain the Benthic Macroinvertebrate Field Data Sheet, Physical

Characterization/Water Quality Field Data Sheet, and sample labels. Example forms are provided in **Attachment 1**.

- Permanent marking pen - used to label samples and to record information in field logbooks and data sheets.
- Sieve Buckets - with 500um mesh. Must have 10 - 12 liter capacity.
- Forceps - to pick organisms from mesh screens and collection nets.
- 95% Ethanol - to preserve samples for analysis.
- Trash Bag - used to dispose of gloves and any other non-hazardous waste generated during sampling.

4.0 METHOD SUMMARY

Benthic macroinvertebrates are collected systematically from all available in-stream habitats by kicking the substrate or jabbing with a D-frame dip net. A total of 20 jabs (or kicks) are taken from all major habitat types in the reach, resulting in sampling approximately 3.1 m^2 of habitat. An organism-based subsample (usually 100, 200, 300, or 500 organisms) is sorted in the laboratory and identified to the lowest practical taxon, generally genus or species.

A quantitative sample is also collected using a surber sampler. Three replicate surber samples are collected from each sampling station to provide a measure of density.

5.0 SAMPLING PROCEDURES

A 100m reach that is representative of the characteristics of the stream should be selected. Whenever possible, the area should be at least 100m upstream from any road or bridge crossing to minimize its effect on stream velocity, depth and overall habitat quality. There should be no major tributaries discharging to the stream in the study area. If a 100m reach is not available for sampling, a standard number of stream widths can be used to measure the stream distance. For example, the EPA's Environmental Monitoring and Assessment Program (EMAP) uses a standard of 40 stream widths for sampling. This approach allows variation in the length of the reach, based on the size of the stream.

Before sampling, complete the physical/chemical field sheet to document site description, weather conditions, and land use. Example forms are provided in **Attachment 1**. After sampling, review this information for accuracy and completeness.

Draw a map of the sampling reach or stream widths on the Field Data Sheet. This map should include in-stream attributes (e.g., riffles, falls, fallen trees, pools, bends, etc.) and important structures, plants, and attributes of the bank and near stream areas. Use an arrow to indicate the direction of flow. Indicate the areas that were sampled for macroinvertebrates, with each sample identification number on the map. Use a hand-held GPS for latitude and longitude determination of the furthest upstream and downstream points of the sampling reach.

5.1 Habitat Types

The following major stream habitat types are colonized by macroinvertebrates and generally support macroinvertebrate diversity in stream ecosystems. Some combination of these habitats will be sampled using this multi-habitat approach to benthic sampling.

Cobble (hard substrate) - In many high-gradient streams, this habitat type will be dominant. Sample shallow areas with coarse (mixed gravel, cobble or larger) substrates by holding the bottom of the dip net against the substrate and dislodging organisms by kicking the substrate for 0.5 m upstream of the net.

Snags - Snags and other woody debris that have been submerged for a relatively long period (not recent deadfall) provide excellent colonization habitat. Sample submerged woody debris by jabbing in medium-sized snag material (sticks and branches). The snag habitat may be kicked first to help dislodge organisms, but only after placing the net downstream of the snag. Accumulated woody material in pool areas are considered snag habitat. Large logs should be avoided because they are generally difficult to sample adequately.

Vegetated banks - When lower banks are submerged and have roots and emergent plants associated with them, they are sampled in a fashion similar to snags. Submerged areas of undercut banks are good habitats to sample. Sample banks with protruding roots and plants by jabbing into the habitat. Bank habitat can be kicked first to help dislodge organisms, but only after placing the net downstream.

Submerged macrophytes - Submerged macrophytes are seasonal in their occurrence and may not be a common feature of many streams, particularly those that are high-gradient. Sample aquatic plants that are rooted on the bottom of the stream in deep water by drawing the net through the vegetation from the bottom to the surface of the water (maximum of 0.5m each jab). In shallow water, sample by bumping or jabbing the net along the bottom in the rooted area, avoiding sediments where possible.

Sand (and other fine sediment) - Usually the least productive macroinvertebrate habitat in streams, this habitat may be the most prevalent in some streams. Sample banks of unvegetated or soft soil by bumping the net along the surface of the substrate rather than dragging the net through soft substrates; this reduces the amount of debris in the sample.

5.2 Sample Collection of Community Samples

5.2.1 General Information

Record the percentage of each habitat type in the reach. Note the sampling gear used, and comment on

conditions of the sampling, e.g., high flows, treacherous rocks, difficult access to stream, or anything that would indicate adverse sampling conditions.

Document observations of aquatic flora and fauna. Make qualitative estimates of macroinvertebrate composition and relative abundance as a cursory estimate of ecosystem health and to check adequacy of sampling.

5.2.2 Non-Wadable Waterbodies

Non-wadable waterbodies should use a Ponar or Ekman dredge to collect sediment-dwelling benthic macroinvertebrates. Sediment collection procedures are similar to the collection techniques discussed in the Sediment Sampling **OU3 SOP No. 5**. Multiple sediment grab samples will be composited and placed in an elutriator bucket or 500um mesh sieve bucket. Add water to the sample and hand-mix gently to break up lumps of sediment. Pour the sample slurry from the tub through the elutriator or sieve bucket which is placed over a second tub to catch the rinse water. Wash the sediment through the mesh with water at very low pressure. Excessive pressure will result in damage to organisms, in particular oligochaetes, and could compromise taxonomic analysis of the sample. Gently agitate the sieve bucket to aid in rinsing the fine sediment out of the sample. It may be necessary to sieve the slurry in small portions to prevent clogging of the mesh. Continue to rinse the composite with surface water until all sediments have been removed, leaving behind any sediment-dwelling invertebrates.

The same volume of sediment should be collected and the same number of rinses should be performed at each sampling location to ensure representativeness between locations. The number of grabs collected and rinses performed should be recorded on the Benthic Macroinvertebrate Field Data Sheet (example provided in **Attachment 1**).

Transfer the sample from the bucket to sample container(s) and preserve in enough 95% ethanol to cover the sample. Forceps may be needed to remove organisms from the bucket. Place a sample identification label that includes date, stream name, sampling location, and collector name into the sample container. The outside of the container should include the same information and the words "preservative: 95% ethanol". If more that one container is needed for a sample, each container label should contain all the information for the sample and should be numbered (e.g., 1 of 2, 2 of 2, etc.). This information will be recorded in the "Sample Log" at the biological laboratory.

Complete the top portion of the Benthic Macroinvertebrate Field Data Sheet (**Attachment 1**).

5.2.3 Wadable Waterbodies

Wadable waterbodies (eg: streams, rivers) can be sampled using a kick-net or dip net. Begin sampling at the downstream end of the reach and proceed upstream. A total of 20 jabs or kicks will be taken over the length of the reach; a single jab consists of forcefully thrusting the net into a productive habitat for a linear distance of 0.5m. A kick is a stationary sampling accomplished by positioning the net and disturbing the substrate for a distance of 0.5m upstream of the net.

Different types of habitat are to be sampled in approximate proportion to their representation of surface area of the total macroinvertebrate habitat in the reach. For example, if snags comprise 50% of the habitat in a reach and riffles comprise 20%, then 10 jabs should be taken in snag material and 4 jabs should be take in riffle areas. The remainder of the jabs (6) would be taken in any remaining habitat type. Habitat types contributing less than 5% of the stable habitat in the stream reach should not be sampled. In this case, allocate the remaining jabs proportionately among the predominant substrates. The number of jabs taken in each habitat type should be recorded on the Benthic Macroinvertebrate Field Data Sheet (example provided in **Attachment 1**).

The jabs or kicks collected from the multiple habitats will be composited to obtain a single homogeneous sample. Every 3 jabs (more often if necessary) wash the collected material by running clean stream water through the net two to three times, being careful to retain the sample inside the net. If clogging does occur, discard the material in the net and redo that portion of the sample in the same habitat type but in a different location. Remove large debris after rinsing and inspecting it for organisms; place any organisms found into the sample container. Do not spend time inspecting small debris in the field.

Transfer the sample from the net to sample container(s) and preserve in enough 95% ethanol to cover the sample. Forceps may be needed to remove organisms from the dip net. Place a sample identification label that includes date, stream name, sampling location, and collector name into the sample container. The outside of the container should include the same information and the words "preservative: 95% ethanol". If more that one container is needed for a sample, each container label should contain all the information for the sample and should be numbered (e.g., 1 of 2, 2 of 2, etc.). This information will be recorded in the "Sample Log" at the biological laboratory.

Complete the top portion of the Benthic Macroinvertebrate Field Data Sheet (**Attachment 1**).

In addition to the sampling of the different habitat types, three surber samples should be collected. Each surber sample will be saved and preserved separately as replicate samples.

5.3 Sample Collection for Analytical Measurements

Sample collection techniques are identical to those utilized to collect benthic community samples. The jabs/kicks or dredge samples collected from multiple habitats will be composited to obtain a single homogeneous sample. Remove any large debris manually and use forceps or elutriation buckets to extract any organisms from the sample; place any organisms found into the appropriate sample container as specified in the QAPP. Continue to collect and composite organisms until the mass requirements for the analytical method are met. Benthic macroinvertebrate samples that will be analyzed for contaminants

should be kept on dry ice.

5.4 Habitat Assessment

Perform habitat assessment after sampling has been completed, and record all observations on the Habitat Assessment Field Data Sheet (**Attachment 1**). Having sampled the various microhabitats and walked the reach helps ensure a more accurate assessment. Conduct the habitat assessment with another team member, if possible.

Return samples to the laboratory and complete the log-in forms (example provided in **Attachment 2**).

6.0 SAMPLE CONTAINERS AND LABELING

Sample labels must be properly completed, including the sample identification code, date, stream name, sampling location, and collector's name and placed into the sample container. The outside of the container should be labeled with the same information. Chain-of-custody forms must include the same information as the sample container labels.

7.0 LABORATORY PROCESSING FOR COMMUNITY SAMPLES

7.1 Laboratory Equipment/Supplies

- log-in sheet for samples (example provided in **Attachment 2**)
- standardized gridded pan (30cm x 36cm) with approximately 30 grids (6cm x 6cm)
- 500 micron sieve
- forceps
- white plastic or enamel pan (15cm x 23cm) for sorting
- specimen vials with caps or stoppers

- sample labels
- benthic macroinvertebrate laboratory bench sheet (example provided in **Attachment 2**)
- dissecting microscope for organism identification
- fiber optics light source
- compound microscope with phase contrast for identification of mounted organisms (e.g., midges)
- 70% ethanol for storage of specimens
- appropriate taxonomic keys

Macroinvertebrate samples should be processed in the laboratory under controlled conditions. Aspects of laboratory processing include subsampling, sorting, and identification of organisms.

All samples should be dated and recorded in the "Sample Log" notebook, which is a three-ring binder book used to store Sample Log forms (**Attachment 2**) upon receipt by laboratory personnel. All information from the sample container label must be included on the sample log sheet. If more than one container was used, the number of containers should be indicated as well. All samples should be sorted in a single laboratory to enhance quality control.

7.2 Subsampling and Sorting

The Rapid Bioassessment Protocol III uses a fixed-count approach to subsampling and sorting the organisms from the sample matrix of detritus, sand, and mud. The following protocol is based on a 200-organism subsample, but it could be used for any subsample size (100, 300, 500, etc.). The subsample is sorted and preserved separately from the remaining sample for quality control checks.

Prior to processing any samples in a lot (i.e., samples within a collection date, specific watershed, or project), complete the sample log-in sheet to verify that all samples have arrived at the laboratory, and are in proper condition for processing.

Thoroughly rinse sample in a 500um mesh sieve to remove preservative and fine sediment. Large organic material (whole leaves, twigs, algal or macrophyte mats, etc.) not removed in the field should be rinsed, visually inspected, and discarded. If the samples have been preserved in alcohol, it will be necessary to soak the sample contents in water for about 15 minutes to hydrate the benthic organisms, which will prevent them from floating on the water surface during sorting. If the sample was stored in more than one container, the contents of all containers for a given sample should be combined at this time. Gently mix the sample by hand while rinsing to make homogeneous.

After washing, spread the sample evenly across a pan marked with grids approximately 6cm x 6cm. On the laboratory bench sheet, note the presence of large or obviously abundant organisms; do not remove them from the pan.

Use a random numbers table to select 4 numbers corresponding to squares (grids) within the gridded pan. Remove all material (organisms and debris) from the four grid squares, and place the material into a shallow white pan and add a small amount of water to facilitate sorting. If there appear (through a cursory count or observation) to be 200 organisms ± 20% (cumulative of 4 grids), then subsampling is complete.

Any organism that is lying over a line separating two grids is considered to be on the grid containing its head. In those instances where it may not be possible to determine the location of the head (worms for instance), the organism is considered to be in the grid containing most of its body.

If the density of organisms is high enough that many more than 200 organisms are contained in the 4 grids, transfer the contents of the 4 grids to a second gridded pan. Randomly select grids for this second level of sorting as was done for the first, sorting grids one at a time until 200 organisms ± 20% are found. If picking through the entire next grid is likely to result in a subsample of greater than 240 organisms, then that grid may be subsampled in the same manner as before to decrease the likelihood of exceeding

240 organisms. That is, spread the contents of the last grid into another gridded pan. Pick grids one at a time until the desired number is reached. The total number of grids for each subsorting level should be noted on the laboratory bench sheet.

7.3 Identification of Macroinvertebrates

Taxonomy can be at any level, but should be done consistently among samples. Genus/species provides more accurate information on ecological/ environmental relationships and sensitivity to impairment. Family level provides a higher degree of precision among samples and taxonomists, requires less expertise to perform, and accelerates assessment results. In either case, only those taxonomic keys that have been peer-reviewed and are available to other taxonomists should be used.

Most organisms are identified to the lowest practical level (generally genus or species) by a qualified taxonomist using a dissecting microscope. Midges (Diptera: Chironomidae) are mounted on slides in an appropriate medium and identified using a compound microscope. Each taxon found in a sample is recorded and enumerated in a laboratory bench notebook and then transcribed to the laboratory bench sheet for subsequent reports. Any difficulties encountered during identification (e.g., missing gills) are noted on these sheets.

Labels with specific taxa names (and the taxonomist's initials) are added to the vials of specimens by the taxonomist. (Note that individual specimens may be extracted from the sample to be included in a reference collection or to be verified by a second taxonomist.) Slides are initialed by the identifying taxonomist. A separate label may be added to slides to include the taxon (taxa) name(s) for use in a voucher or reference collection.

Record the identity and number of organisms on the Laboratory Bench Sheet (**Attachment 2**). Either a tally counter or "slash" marks on the bench sheet can be used to keep track of the cumulative count. Also, record the life stage of the organisms, the taxonomist's initials and the Taxonomic Certainty Rating (TCR)

as a measure of confidence.

In the spaces provided on the bench sheet, explain certain TCR ratings or condition of organisms. Other comments can be included to provide additional insights for data interpretation. If QC was performed, record on the back of the bench sheet.

For archiving samples, specimen vials, (grouped by station and date), are placed in jars with a small amount of denatured 70% ethanol and tightly capped. The ethanol level in these jars must be examined periodically and replenished as needed, before ethanol loss from the specimen vials takes place. A stick-on label is placed on the outside of the jar indicating sample identifier, date, and preservative (denatured 70% ethanol).

All samples should be stored on wet ice (4°C) in a secured cooler. Ship samples under chain- of-custody, protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

8.0 SUBSAMPLE PROCEDURE MODIFICATIONS

As an improvement to the mechanics of the technique, a sorting tray was designed that consists of two parts, a rectangular plastic or plexiglass pan (36cm x 30cm) with a rectangular sieve insert. The sample is placed on the sieve, in the pan and dispersed evenly.

When a random grid(s) is selected, the sieve is lifted to temporarily drain the water. A "cookie-cutter" like metal frame 6cm x 6cm is used to clearly define the selected grid; debris overhanging the grid may be cut with scissors. A 6cm flat scoop is used to remove all debris and organisms from the grid. The contents are then transferred to a separate sorting pan with water for removal of macroinvertebrates.

These modifications have allowed for rapid isolation of organisms within the selected grids and easy

removal of all organisms and debris within a grid while eliminating investigator bias. Save the sorted debris residue in a separate container. Add a label that includes the words "sorted residue" in addition to all prior sample label information and preserve in 95% ethanol. Save the remaining unsorted sample debris residue in a separate container labeled "sample residue"; this container should include the original sample label. Length of storage and archival is determined by the laboratory or benthic section supervisor.

Place the sorted 200-organism (± 20%) subsample into glass vials, and preserve in 70% ethanol. Label the vials inside with the sample identifier or lot number, date, stream name, sampling location and taxonomic group. If more than one vial is needed, each should be labeled separately and numbered (e.g., 1 of 2, 2 of 2). For convenience in reading the labels inside the vials, insert the labels left-edge first. If identification is to occur immediately after sorting, a petri dish or watch glass can be used instead of vials.

Midge (Chironomidae) larvae and pupae should be mounted on slides in an appropriate medium (e.g., Euperal, CMC-9); slides should be labeled with the site identifier, date collected, and the first initial and last name of the collector. As with midges, worms (Oligochaeta) must also be mounted on slides and should be appropriately labeled.

Fill out header information on Laboratory Bench Sheet (see **Attachment 2**). Also check subsample target number. Complete back of sheet for subsampling/sorting information. Note number of grids picked, time expenditure, and number of organisms. If QC check was performed on a particular sample, person conducting QC should note findings on the back of the Laboratory Bench Sheet. Calculate sorting efficiency to determine whether sorting effort passes or fails.

Record date of sorting and slide monitoring, if applicable, on Log-In Sheet as documentation of progress and status of completion of sample lot.

9.0 DECONTAMINATION

After sampling has been completed at a given site, all nets, pans, etc. that have come in contact with the sample should be rinsed thoroughly, examined carefully, and picked free of organisms or debris. Any additional organisms found should be placed into the sample containers. The equipment should be examined again prior to use at the next sampling site.

Excess sediment and substrate material not included in the sample should be washed into the stream, pond, lake, or surface impoundment where it came from. All marker flags (if reused) should be decontaminated by wiping off with towels and/or baby wipes before re-use.

Throw all used wipes and gloves into the trash bags and take with you to dispose of at the field office.

10.0 RECORD KEEPING AND QUALITY CONTROL

Each field crew will carry a three-ring binder book that contains the Benthic Macroinvertebrate Field Data Sheet, Physical Characterization/Water Quality Field Data Sheet, and sample labels. In addition, a field notebook should be maintained by each individual or team that is collecting samples, as described in the Project Plan. Each sampling location must be recorded on the site diagram. Each sample should have an ID number affixed to the outside of the wide-mouth bottle, and the duplicate label must be affixed to the sample data sheet. Deviations from this sampling plan should be noted in the field notebook, as necessary.

10.1 Required Information

For each location, the notebook information must include:

a. date
b. time
c. personnel

d. weather conditions

e. sample identification numbers that were used

f. descriptions of any deviations to the Project Plan and the reason for the deviation

Samples taken from waters with visible color abnormalities, foaming, unusual odor, iridescent film, or other indications of non-homogeneous conditions should also be noted. Field personnel will collect the proper type and quantity of quality control samples as prescribed in the QAPP.

10.2 Field Quality Control Samples

The type of quality control samples, and the frequency of collection, are specified in the QAPP. The following quality control samples will be collected.

Field Duplicate: Field duplicate samples are collected at the same time as the primary sample, and are used to evaluate precision and reproducibility of the analysis and sampling technique or collection team. In this case, the field duplicate sample is a second sample of benthic macroinvertebrates collected from the same reach or widths of a stream.

11.0 REFERENCES

Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling. 1999. Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers: Periphyton, Benthic Macroinvertebrates and Fish, Second Edition. EPA 841-B-99-002. U.S. Environmental Protection Agency; Office of Water; Washington, D.C.

USEPA. 2003. Sampling and Analytical Procedures for GLNPO's Open Lake Water Quality Survey of the Great Lakes; Chapter 4 - Biological Parameters; LG406 - Standard Operating Procedure for Benthic Invertebrate Field Sampling Procedure, Revision 07. EPA 905-R-03-002. March 2003.

ATTACHMENT 1

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Benthic Macroinvertebrate Field Data Sheet

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BENTHIC MACROINVERTEBRATE FIELD DATA SHEET

STREAM NAME	LOCATION	
STATION # RIVERMILE	STREAM CLASS	
LAT LONG	RIVER BASIN	
STORET #	AGENCY	
INVESTIGATORS		LOT NUMBER
FORM COMPLETED BY	DATE _____ TIME ____ AM PM	REASON FOR SURVEY

HABITAT TYPES	**Indicate the percentage of each habitat type present** ❑ Cobble____% ❑ Snags____% ❑ Vegetated Banks____% ❑ Sand____% ❑ Submerged Macrophytes____% ❑ Other ( )____%
SAMPLE COLLECTION	**Gear used** ❑ D-frame ❑ kick-net ❑ Other _____ **How were the samples collected?** ❑ wading ❑ from bank ❑ from boat **Indicate the number of jabs/kicks taken in each habitat type.** ❑ Cobble____ ❑ Snags____ ❑ Vegetated Banks____ ❑ Sand____ ❑ Submerged Macrophytes____ ❑ Other ( )____
GENERAL COMMENTS	

QUALITATIVE LISTING OF AQUATIC BIOTA
Indicate estimated abundance: 0 = Absent/Not Observed, 1 = Rare, 2 = Common, 3= Abundant, 4 = Dominant

Periphyton	0	1	2	3	4	Slimes	0	1	2	3	4
Filamentous Algae	0	1	2	3	4	Macroinvertebrates	0	1	2	3	4
Macrophytes	0	1	2	3	4	Fish	0	1	2	3	4

FIELD OBSERVATIONS OF MACROBENTHOS
Indicate estimated abundance: 0 = Absent/Not Observed, 1 = Rare (1-3 organisms), 2 = Common (3-9 organisms), 3= Abundant (>10 organisms), 4 = Dominant (>50 organisms)

Porifera	0	1	2	3	4	Anisoptera	0	1	2	3	4	Chironomidae	0	1	2	3	4
Hydrozoa	0	1	2	3	4	Zygoptera	0	1	2	3	4	Ephemeroptera	0	1	2	3	4
Platyhelminthes	0	1	2	3	4	Hemiptera	0	1	2	3	4	Trichoptera	0	1	2	3	4
Turbellaria	0	1	2	3	4	Coleoptera	0	1	2	3	4	Other	0	1	2	3	4
Hirudinea	0	1	2	3	4	Lepidoptera	0	1	2	3	4						
Oligochaeta	0	1	2	3	4	Sialidae	0	1	2	3	4						
Isopoda	0	1	2	3	4	Corydalidae	0	1	2	3	4						
Amphipoda	0	1	2	3	4	Tipulidae	0	1	2	3	4						
Decapoda	0	1	2	3	4	Empididae	0	1	2	3	4						
Gastropoda	0	1	2	3	4	Simuliidae	0	1	2	3	4						
Bivalvia	0	1	2	3	4	Tabinidae	0	1	2	3	4						
						Culcidae	0	1	2	3	4						

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Physical Characterization/Water Quality Field Data Sheet

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PHYSICAL CHARACTERIZATION/WATER QUALITY FIELD DATA SHEET
(Pg. 1)

STREAM NAME	LOCATION	
STATION #_____ RIVERMILE_____	STREAM CLASS	
LAT _____ LONG _____	RIVER BASIN	
STORET #	AGENCY	
INVESTIGATORS		
FORM COMPLETED BY	DATE _____ TIME _____ AM PM	REASON FOR SURVEY

WEATHER CONDITIONS

Now		Past 24 hours
❑	storm (heavy rain)	❑
❑	rain (steady rain)	❑
❑	showers (intermittent)	❑
____%❑	%cloud cover	____%
❑	clear/sunny	❑
		❑

Has there been a heavy rain in the last 7 days? ❑ Yes ❑ No

Air Temperature____°C

Other_____

SITE LOCATION AND MAP

Draw a map of the site and indicate the areas sampled (or attach a photograph)

STREAM CHARACTERIZATION

Stream Subsystem
❑ Perennial ❑ Intermittent ❑ Tidal

Stream Origin
❑ Glacial
❑ Non-glacial montane
❑ Swamp and bog
❑ Spring-fed
❑ Mixture of origins
❑ Other_____

Stream Type
❑ Coldwater ❑ Warmwater

Catchment Area_____km^2

PHYSICAL CHARACTERIZATION/WATER QUALITY FIELD DATA SHEET
(Pg. 2)

WATERSHED FEATURES	**Predominant Surrounding Landuse** ❑ Forest ❑ Commercial ❑ Field/Pasture ❑ Industrial ❑ Agricultural ❑ Other _____ ❑ Residential **Local Watershed NPS Pollution** ❑ No evidence ❑ Some potential sources ❑ Obvious sources **Local Watershed Erosion** ❑ None ❑ Moderate ❑ Heavy
RIPARIAN VEGETATION (18 meter buffer)	**Indicate the dominant type and record the dominant species present** ❑ Trees ❑ Shrubs ❑ Grasses ❑ Herbaceous **dominant species present** _____
INSTREAM FEATURES	**Estimated Reach Length** _____m **Estimated Stream Width** _____m **Sampling Reach Area** _____m^2 **Area in km^2 (m^2x1000)** _____km^2 **Estimated Stream Depth** _____m **Surface Velocity (at thalweg)** _____m/sec **Canopy Cover** ❑ Partly open ❑ Partly shaded ❑ Shaded **High Water Mark** _____m **Proportion of Reach Represented by Stream Morphology Types** ❑ Riffle_____% ❑ Run_____% ❑ Pool_____% **Channelized** ❑ Yes ❑ No **Dam Present** ❑ Yes ❑ No
LARGE WOODY DEBRIS	**LWD** _____m^2 **Density of LWD** _____m^2/km^2 **(LWD/ reach area)**
AQUATIC VEGETATION	**Indicate the dominant type and record the dominant species present** ❑ Rooted emergent ❑ Rooted submergent ❑ Rooted floating ❑ Free floating ❑ Floating Algae ❑ Attached Algae **dominant species present** _____ **Portion of the reach with aquatic vegetation** _____%
WATER QUALITY	**Temperature**_____^{0} C **Specific Conductance**_____ **Dissolved Oxygen** _____ **pH** _____ **Turbidity** _____ **WQ Instrument Used** _____ **Water Odors** ❑ Normal/None ❑ Sewage ❑ Petroleum ❑ Chemical ❑ Fishy ❑ Other_____ **Water Surface Oils** ❑ Slick ❑ Sheen ❑ Globs ❑ Flecks ❑ None ❑ Other_____ **Turbidity (if not measured)** ❑ Clear ❑ Slightly turbid ❑ Turbid ❑ Opaque ❑ Stained ❑ Other_____
SEDIMENT/ SUBSTRATE	**Odors** ❑ Normal ❑ Sewage ❑ Petroleum ❑ Chemical ❑ Anaerobic ❑ None ❑Other_____ **Oils** ❑ Absent ❑ Slight ❑ Moderate ❑ Profuse **Deposits** ❑ Sludge ❑ Sawdust ❑ Paper fiber ❑ Sand ❑ Relict shells ❑ Other_____ **Looking at stones which are not deeply embedded, are the undersides black in color?** ❑ Yes ❑ No

PHYSICAL CHARACTERIZATION/WATER QUALITY FIELD DATA SHEET
(Pg. 3)

INORGANIC SUBSTRATE COMPONENTS (should add up to 100%)			ORGANIC SUBSTRATE COMPONENTS (does not necessarily add up to 100%)		
Substrate Type	**Diameter**	**% Composition in Sampling Reach**	**Substrate Type**	**Characteristic**	**% Composition in Sampling Area**
Bedrock			Detritus	sticks, wood, coarse plant materials (CPOM)	
Boulder	> 256 mm (10")				
Cobble	64-256 mm (2.5"-10")		Muck-Mud	black, very fine organic (FPOM)	
Gravel	2-64 mm (0.1"-2.5")				
Sand	0.06-2mm (gritty)		Marl	grey, shell fragments	
Silt	0.004-0.06 mm				
Clay	< 0.004 mm (slick)				

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Habitat Assessment Field Data Sheet

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HABITAT ASSESSMENT FIELD DATA SHEET - LOW GRADIENT STREAMS

STREAM NAME	LOCATION	
STATION #_____ RIVERMILE_____	STREAM CLASS	
LAT _____ LONG _____	RIVER BASIN	
STORET #	AGENCY	
INVESTIGATORS		
FORM COMPLETED BY	DATE _____ TIME _____ AM PM	REASON FOR SURVEY

Parameters to be evaluated in sampling reach	Habitat Parameter	Condition Category			
		Optimal	**Suboptimal**	**Marginal**	**Poor**
	1. Epifaunal Substrate/ Available Cover	Greater than 50% of substrate favorable for epifaunal colonization and fish cover; mix of snags, submerged logs, undercut banks, cobble or other stable habitat and at stage to allow full colonization potential (i.e., logs/snags that are not new fall and not transient).	30-50% mix of stable habitat; well-suited for full colonization potential; adequate habitat for maintenance of populations; presence of additional substrate in the form of newfall, but not yet prepared for colonization (may rate at high end of scale).	10-30% mix of stable habitat; habitat availability less than desirable; substrate frequently disturbed or removed.	Less than 10% stable habitat; lack of habitat is obvious; substrate unstable or lacking.
	SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
	2. Pool Substrate Characterization	Mixture of substrate materials, with gravel and firm sand prevalent; root mats and submerged vegetation common.	Mixture of soft sand, mud, or clay; mud may be dominant; some root mats and submerged vegetation present.	All mud or clay or sand bottom; little or no root mat; no submerged vegetation.	Hard-pan clay or bedrock; no root mat or vegetation.
	SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
	3. Pool Variability	Even mix of large-shallow, large-deep, small-shallow, small-deep pools present.	Majority of pools large-deep; very few shallow.	Shallow pools much more prevalent than deep pools.	Majority of pools small-shallow or pools absent.
	SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
	4. Sediment Deposition	Little or no enlargement of islands or point bars and less than <20% of the bottom affected by sediment deposition.	Some new increase in bar formation, mostly from gravel, sand or fine sediment; 20-50% of the bottom affected; slight deposition in pools.	Moderate deposition of new gravel, sand or fine sediment on old and new bars; 50-80% of the bottom affected; sediment deposits at obstructions, constrictions, and bends; moderate deposition of pools prevalent.	Heavy deposits of fine material, increased bar development; more than 80% of the bottom changing frequently; pools almost absent due to substantial sediment deposition.
	SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
	5. Channel Flow Status	Water reaches base of both lower banks, and minimal amount of channel substrate is exposed.	Water fills >75% of the available channel; or <25% of channel substrate is exposed.	Water fills 25-75% of the available channel, and/or riffle substrates are mostly exposed.	Very little water in channel and mostly present as standing pools.
	SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0

Parameters to be evaluated broader than sampling reach

Habitat Parameter	Condition Category			
	Optimal	**Suboptimal**	**Marginal**	**Poor**
6. Channel Alteration	Channelization or dredging absent or minimal; stream with normal pattern.	Some channelization present, usually in areas of bridge abutments; evidence of past channelization, i.e., dredging, (greater than past 20 yr) may be present, but recent channelization is not present.	Channelization may be extensive; embankments or shoring structures present on both banks; and 40 to 80% of stream reach channelized and disrupted.	Banks shored with gabion or cement; over 80% of the stream reach channelized and disrupted. Instream habitat greatly altered or removed entirely.
SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
7. Channel Sinuosity	The bends in the stream increase the stream length 3 to 4 times longer than if it was in a straight line. (Note - channel braiding is considered normal in coastal plains and other low-lying areas. This parameter is not easily rated in these areas.)	The bends in the stream increase the stream length 1 to 2 times longer than if it was in a straight line.	The bends in the stream increase the stream length 1 to 2 times longer than if it was in a straight line.	Channel straight; waterway has been channelized for a long distance.
SCORE	20 19 18 17 16	15 14 13 12 11	10 9 8 7 6	5 4 3 2 1 0
8. Bank Stability (score each bank)	Banks stable; evidence of erosion or bank failure absent or minimal; little potential for future problems. <5% of bank affected.	Moderately stable; infrequent, small areas of erosion mostly healed over. 5-30% of bank in reach has areas of erosion.	Moderately unstable; 30-60% of bank in reach has areas of erosion; high erosion potential during floods.	Unstable; many eroded areas; "raw" areas frequent along straight sections and bends; obvious bank sloughing; 60-100% of bank has erosional scars.
SCORE ___ (LB)	Left Bank 10 9	8 7 6	5 4 3	2 1 0
SCORE ___ (RB)	Right Bank 10 9	8 7 6	5 4 3	2 1 0
9. Vegetative Protection (score each bank) Note: determine left or right side by facing downstream.	More than 90% of the streambank surfaces and immediate riparian zone covered by native vegetation, including trees, understory shrubs, or nonwoody macrophytes; vegetative disruption through grazing or mowing minimal or not evident; almost all plants allowed to grow naturally.	70-90% of the streambank surfaces covered by native vegetation, but one class of plants is not well-represented; disruption evident but not affecting full plant growth potential to any great extent; more than one-half of the potential plant stubble height remaining.	50-70% of the streambank surfaces covered by vegetation; disruption obvious; patches of bare soil or closely cropped vegetation common; less than one-half of the potential plant stubble height remaining.	Less than 50% of the streambank surfaces covered by vegetation; disruption of streambank vegetation is very high; vegetation has been removed to 5 centimeters or less in average stubble height.
SCORE ___ (LB)	Left Bank 10 9	8 7 6	5 4 3	2 1 0
SCORE ___ (RB)	Right Bank 10 9	8 7 6	5 4 3	2 1 0
10. Riparian Vegetative Zone Width (score each bank riparian zone)	Width of riparian zone >18 meters; human activities (i.e., parking lots, roadbeds, clear-cuts, lawns, or crops) have not impacted zone.	Width of riparian zone 12-18 meters; human activities have impacted zone only minimally.	Width of riparian zone 6-12 meters; human activities have impacted zone a great deal.	Width of riparian zone <6 meters: little or no riparian vegetation due to human activities.
SCORE ___ (LB)	Left Bank 10 9	8 7 6	5 4 3	2 1 0
SCORE ___ (RB)	Right Bank 10 9	8 7 6	5 4 3	2 1 0

Total Score _____

ATTACHMENT 2

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Sample Log-in Sheet

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BENTHIC MACROINVERTEBRATE SAMPLE LOG-IN SHEET										
Date Collected	Collected By	Number of Containers	Preservation	Station #	Stream Name and Location	Date Received by Lab	Lot Number	Date of Completion		
								sorting	mounting	identification

Benthic Macroinvertebrate Laboratory Bench Sheet

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BENTHIC MACROINVERTEBRATE LABORATORY BENCH SHEET (Pg. 1)

page _____ of _____

STREAM NAME	LOCATION
STATION #_____ RIVERMILE_____	STREAM CLASS
LAT _____ LONG _____	RIVER BASIN
STORET #	AGENCY
COLLECTED BY DATE_____	LOT #
TAXONOMIST DATE_____	SUBSAMPLE TARGET ❑ 100 ❑ 200 ❑ 300 ❑ Other ____

Enter Family and/or Genus and Species name on blank line.

Organisms		No.	LS	TI	TCR	Organisms		No.	LS	TI	TCR
Oligochaeta						Megaloptera					
Hirudinea						Coleoptera					
Isopoda											
Amphipoda						Diptera					
Decapoda											
Ephemeroptera											
						Gastropoda					
						Pelecypoda					
Plecoptera											
						Other					
Trichoptera											
Hemiptera											

Taxonomic certainty rating (TCR) 1-5:1=most certain, 5=least certain. If rating is 3-5, give reason (e.g., missing gills).

LS= life stage: I = immature; P = pupa; A = adult TI = Taxonomists initials

Total No. Organisms _____ **Total No. Taxa** _____

BENTHIC MACROINVERTEBRATE LABORATORY BENCH SHEET (Pg. 2)

SUBSAMPLING/SORTING INFORMATION

Sorter _____

Date _____

Number of grids picked: _____

Time expenditure _____ No. of organisms _____

Indicate the presence of large or obviously abundant organisms:

QC: ❑ YES ❑ NO **QC Checker** _____

# organisms originally sorted		(# organisms recovered by checker	+	# organisms originally sorted)	=	% sorting efficiency
	÷					

≥90%, sample passes _____

<90%, sample fails, action taken _____

TAXONOMY

ID _____

Date _____

Explain TCR ratings of 3-5:

Other Comments (e.g. condition of specimens):

QC: ❑ YES ❑ NO **QC Checker** _____

Organism recognition ❑ pass ❑ fail
Verification complete ❑ YES ❑ NO

Date: May 27, 2008 SOP MAMMAL-LIBBY-OU3 (Rev. 0)

Title: SMALL MAMMAL COLLECTION AND PROCESSING

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	_____	_____
SOP Author	_____	_____

Revision Number	Date	Reason for Revision
0	05/27/2008	--
1		

CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for collection of small mammals for biological surveys, chemical analysis of tissues and/or histopathological examination. This procedure will be used by USEPA Region 8 for the Remedial Investigation work for Operable Unit 3 performed at the Libby Asbestos Superfund site.

This document focuses on methods and equipment that are readily available and typically applied in collecting small mammals. It is not intended to provide an all-inclusive discussion of small mammal collection methods. Specific sampling problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be clearly described in the project-specific sampling plan and approved by the Project Manager and the Quality Manager.

2.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects site-specific sampling methods, sample locations, and constituents to be analyzed with input from other key project staff.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Biologist: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL, field biologist, or engineer in the implementation of tasks. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

3.0 EQUIPMENT

3.1 Organizational and Safety Equipment

- Sampling and Analysis Plan (SAP) and Quality Assurance Project Plan (QAPP)
- Health and Safety Plan (HASP)
- Safety equipment (e.g., Tyvek, respirators with high-efficiency particulate air (HEPA) filters, surgical gloves, nitrile gloves, eye protection, first aid kit)
- Clipboard, writing utensils, permanent waterproof ink marker
- Site maps
- Global Positioning System (GPS) navigation/survey equipment
- Digital camera

3.2 Trap Setting and Data Recording Equipment

- Data collection sheets and field log books
- Spring loaded scale
- Tape measure, 100-foot length
- Survey flags and flagging tape
- Leather gloves
- Sherman Live Traps
- Bait (e.g., oats, peanut butter)

3.3 Sample Processing and Shipment Equipment

- Nitrile gloves
- Surgical gloves
- Wet ice
- Dry ice
- Storage cooler
- Small/large resealable plastic bags
- Plastic jars
- Garbage bags
- Duct tape

3.4 Decontamination Equipment

- 5-gallon plastic buckets
- Hypochlorite bleach or Lysol™ disinfecting solution
- Scrub brushes
- Paper towels
- Garbage bags

4.0 SAMPLE COLLECTION

4.1 Preparation

A scientific collection permit should be obtained from the appropriate federal or state agency. Most states have permit information available on the internet. A natural heritage search for threatened or endangered species should also be requested from the state. In addition, permission from the landowner(s) must be received prior to trapping at the site or reference areas.

4.2 Sampling Location Selection

Four areas are identified for small mammal trapping. These locations are listed in the following table along with the rationale for their selection. The exact locations of the sampling areas and placement of trap lines will be made during the initial field reconnaissance based on the identified habitats, terrain, access and other considerations.

Location ID	General Descriptions and Rationale	General Identified Areas
SMT-1	Area on the Mine Site Disturbed Area. This area is expected to have the highest asbestos exposures but not the best habitat to support species.	MW-6 or MW-16
SMT-2	Near the disturbed Mine Site Area in an area with better habitat than SMT-1 (meadow/forested) with known asbestos contamination in soils, tree bark and duff.	Near SL45-01
SMT-3	Riparian area near water body and known asbestos contamination in sediments and/or surface water.	Tailings Pond
SMT-Ref	Reference area with habitat matched closely in terms of vegetative cover and elevation to SMT-2.	Area upwind of OU3 to the west

4.3 Targeted Species

The targeted mammalian species for collection in the mined area and forested area are the ground foraging species (invertivore, herbivore, omnivore). The targeted species in the riparian area are aquatic invertivores and omnivores. Any protected state or federal species captured will be released. The following table provides the list of mammalian wildlife Exposed Receptor Groups for Libby OU3 and possible species within each group.

Wildlife Exposed Receptor Groups Identified in SAP			
Exposed Receptor Group		**Description of Exposed Group**	**Species in Group Common name *(Genus species)*-Number Reported[1]**
Mammalian	Ground Invertivore	Mammalian invertivorous species that feed primarily on soil invertebrates, forage on the ground and may inhabit underground burrows.	Dusky or Montane Shrew (*Sorex monticolus*) - 7 Masked Shrew (*Sorex cinereus*) - 16 Pygmy Shrew (*Sorex hoyi*) - 4 Vagrant Shrew (*Sorex vagrans*) – 39
	Arboreal Invertivore	Mammalian invertivorous and omnivorous species that feed primarily in trees.	Northern Flying Squirrel (*Glaucomys sabrinus*) - Red-tailed Chipmunk (*Tamias ruficaudus*) -
	Ground Herbivore/ Omnivore	Mammalian herbivorous species that feed primarily on plant material, forage on the ground and may inhabit burrows or nests on the ground.	Bushy-tailed Woodrat (*Neotoma cinerea*) - 4 Columbian Ground Squirrel (*Spermophilus columbianus*) - 12 Deer Mouse (*Peromyscus maniculatus*) - 60 Golden-mantled Ground Squirrel (*Spermophilus lateralis*) -2 Heather Vole (*Phenacomys intermedius*) - 15 Hoary Marmot (*Marmota caligata*) - 12 Long-tailed Vole (*Microtus longicaudus*) - 13 Mountain Cottontail (*Sylvilagus nuttallii*) Northern Pocket Gopher (*Thomomys talpoides*) - 1 Pika (*Ochotona princeps*) - 12 Red Squirrel (*Tamiasciurus hudsonicus*) - 19 Southern Red-backed Vole (*Clethrionomys gapperi*) - 35 Snowshoe Hare (*Lepus americanus*) -1 Yellow-bellied Marmot (*Marmota flaviventris*) - 3 Yellow pine chipmunk (*Tamias amoenus*) - 10 Western Jumping Mouse (*Zapus princeps*) – 17
	Aquatic Invertivore /Omnivore	Mammalian species that feed in riparian areas on aquatic plants and aquatic invertebrates	Water Shrew (*Sorex palustris*) Water Vole (*Microtus richardsoni*) – 4

[1]Number of occurrences in Lincoln, County Montana (http://fieldguide.mt.gov/)
If number is not presented, the data were not available.

In nine west-central Montana forest stands (five dominated by old-growth ponderosa pine (*Pinus ponderosa*) and four by western larch (*Larix occidentalis*) over 22,752 trap nights, the most commonly collected species were deer mice (*Peromyscus maniuclatus*), southern red-backed voles (*Clethrionomys gapperi*), and red-tailed chipmunks (*Tamias ruficaudus*) (Pearson and Ruggiero, 2003). Yellowpine chipmunk (*Tamias amoenus*), golden-mantled ground squirrel (*Spermophilus lateralis*), vagrant shrew (*Sorex vagrans*), dusky or montane shrew (*Sorex monticolus*), snowshoe hare (*Lepus americanus*) and red squirrel (*Tamiasciurus hudsonicus*) were also collected but less frequently (Pearson and Ruggiero, 2003). This information agrees with the reported frequency of sightings of ground dwelling small mammalian species as reported in the Montana Tracker (see preceding table). The most common ground herbivore/omnivore reported in Lincoln county are the deer mouse and the southern red-backed vole which are the two most common species captured in the trapping completed by Pearson and Ruggiero (2003). This agreement provides an indication of what species to expect to be trapped using line transect trapping and Sherman traps at Libby OU3.

4.4 Trap Selection

While many types of traps are available for the collection of small mammals, this small mammal collection SOP will use Sherman Live traps. Sherman Live traps are a type of box trap that are the most effective for capturing small terrestrial mammals unharmed (Wilson et al., 1996). As shown in Figure 1, this trap is rectangular in shape with a spring-loaded door that becomes triggered once an animal enters the trap. Box traps are recommended over simple snap traps due to reduced occurrences of predation and trap disturbance by raccoons and deer. Snap traps are lightweight and easily triggered or moved by non-target species. In addition, once an animal is captured in a snap trap, it becomes a likely target for predation. The heavier box trap, with solid sides, is better suited to withstand disruption by predation. Live trapping is also preferred for the collection of samples for histopathology examination. Animals collected from kill traps may decompose prior to collection making tissue examination impossible. The Sherman Live traps come in a variety of sizes. The size to be used for the Libby OU3 effort is 8 X 9 X 23cm).

4.5 Trap Placement

Methods for capturing mammals and in particular the use of trap arrays are reviewed by Wilson et al., 1996. Typical methods of trap placement include transects, grids and webs Pearson and Ruggiero (2003) compared transect versus grid trapping arrangements for sampling small mammal communities in two forest cover types in west central Montana. They found that transect arrangements compared to grid arrangements yield more total captures, more individual captures and more species than grid arrangements in both cover types in both of the years examined. Differences between the two methods were greatest when small mammals were least abundant. Based on this reported efficiency and the lower level of effort required for the line transect method compared to the grid method, the line transect trap method will be used to collect small mammals at Libby OU3.

In the line transect method, traps are placed at equal intervals along a line which is located randomly within a habitat type. More than one line may be located within a habitat type (sampling location). Traps should be placed at habitat features (e.g., log, tree, runway, burrow) as long as they lie within 2 meters of the point. Wilson et al. (1996) recommends placing two traps at each trap point to avoid the saturation of traps with “trap-happy” individuals that are readily captured. The practice increases the chances that animals that are less active or less attracted to traps will be caught.

For Libby OU3, transect trapping lines will be used to determine the placement of traps, as this method ensures the highest rate of mammal trapping success (Wilson et al., 1996). Transect lines will be approximately 150 meters (m) in length with traps placed approximately every 10 m along each transect line. There will be three transect trapping lines per sampling location.

Trap lines should be numbered or lettered sequentially. Each individual trap along the trap line should also be assigned a number, based on its position along the line. The

location and orientation of each trap line should be sketched in the field logbook and also recorded on either a site map or an aerial photo. The start and end of each grid line or trap line should be marked with a survey flag and/or length of flagging tape tied to a branch at eye level. The flag or flagging should be labeled with the trap area, trap line, and trap number, using a thick waterproof marker. In heavily vegetated areas, individual trap locations may also be marked with a labeled survey flag. This simplifies trap relocation and reduces habitat destruction during subsequent trap checks. Flags should be placed so that they do not impede an animal's progress toward the trap. Traps should seldom be set in open areas, since small mammals usually avoid these areas due to the increased likelihood of predation. Success can still be increased by placing traps along fallen logs, large roots, or in brushy areas. However, traps should be placed so that the release is not impeded by vegetation or other obstructions.

4.5 Trapping Effort

Trapping effort is the product of the number of traps used and the time over which those traps are monitored. The number of traps multiplied by the number of "trap-nights" gives the number of "trap-nights" for a particular study. Wilson et al. (1996) recommends a minimum of 500 trap nights for a preliminary investigation of a habitat. Data from studies with similar trapping effort can be compared using relatively simple models that include capture indices and abundance indices.

Wilson et al. (1996) recommends a trap transect be at least 150 m long with traps placed every 10 to 15 m. A general rule is to space traps at a distance no greater than the radius of a circle having an area equal to that of the average home range (if known) of the target species. The deer mouse is the most likely organism to be collected based on the data evaluated in the Problem Formulation (USEPA, 2008). This species has a reported home range averaging one hectare or less and may range from a few hundred to a few thousand square meters (http://www.natureserve.org/). Based on this information of trap spacing of 10 meters is more than adequate for a 200 square meter home range.

The targeted trapping effort at Libby OU3 will be 450 trap nights for the Phase IIC SAP. Three 150 m line transects will be established at each of the sampling locations and traps placed (2 each) at 10 m intervals and collected over a five day period of time. This design will result in a 450 trap night effort per sampling location. The trapping effort (time) required to complete a species inventory can be determined with a species accumulation curve, a plot of cumulative number of species captured versus cumulative trapping effort. When the curve reaches a plateau, or when the capture of species or individuals no longer increases with additional effort, the trapping effort may be adequate.

4.6 Trap Setting and Baiting

Traps will be set in place and baited, but rendered non-functional for 6 days prior to the trapping event. This period of time will allow the animals to become accustomed to the traps and will decrease "trap shyness" (Wilson et al., 1996).

Traps will be set at dusk and checked after the first 2 hours of sunlight in order to capture diurnal (active in the daytime), nocturnal, and corpuscular (active at dusk and dawn) animals. Traps will be baited when they are set. Bait should be carried in a resealable plastic bag and dispensed as needed. The bait will consist of a mixture of 50:50 peanut butter and rolled oats. [Note: The relative proportions of each can be modified to suit field conditions (e.g., use less peanut butter in warmer weather)]. Traps should be baited so that the bait does not fall off. Local invertebrates and/or earthworms may be added to the trap bait to increase the chance of attracting shrews. Traps will be set and checked for 5 consecutive days or until the target numbers of organisms are collected.

4.7 Trap Checks and Data Recording

A field team of two people will be used to check the traps, handle and capture the animals, and record the necessary information. For each trap where an animal is captured, the date, time, and trap identification number will be recorded on a Field Small Mammal Trap Log Form (Appendix A). For the purposes of the Libby OU3 Phase IIC investigation, sample IDs for mammal trapping will be labeled as follows:

SMT-__-x-y-z

where:

• SMT__ is the sampling location number
• x is the unique trap transect line number
• y is the unique location on the transect line
• z is the unique small mammal identification number (e.g., if 2 mammals are captured at the same trap on different days, the z value for the second mammal caught is 2).

The animal will be identified to the species level, classified as alive or dead, and any physical abnormalities will be recorded. Each captured mammal will be photographed. When a sprung Sherman Live trap is located, it will be carefully picked up, and the trap door will be depressed to check for captured animals. If the trap contains an animal, the handler will cover the trap door with a plastic zip lock bag and gently shake the animal into the bag. If no animal is discovered inside the trap, “empty sprung trap” (EST) or “no capture” (NC) will be recorded on the data sheet, as appropriate. The trap will then be placed back in the “unarmed” position on the ground until dusk and then reset and rebaited.

All personnel performing trap checks should wear appropriate personal protective equipment as specified in the site-specific Health and Safety Plan (HASP). At a minimum protection should include surgical gloves underneath an exterior pair of leather or thick rubber gloves (to prevent the interior gloves from getting torn on the sharp surfaces of the traps) and half face respirators fitted with HEPA filters. When checking traps in dry or dusty conditions, full-face respirators with HEPA filters (or half-face

respirators with appropriate eye protection) should be worn, along with disposable coveralls (e.g., tyvek).

4.8 Measurements on Trapped Mammals

The following information will be recorded for each of the mammals trapped. Data is recorded on the small mammal trapping log sheet. A separate log sheet is maintained for each trapping location over the duration of the trapping event.

Species

The species is recorded by a common name abbreviation. Abbreviations are listed in the following table.

Exposed Receptor Group		Common name	
Mammalian	Ground Invertivore	Dusky or Montane Shrew	DSKS
		Masked Shrew	MSKS
		Pygmy Shrew	PYGS
		Vagrant Shrew	VAGS
	Arboreal Invertivore	Northern Flying Squirrel	NFSQ
		Red-tailed Chipmunk	RTCM
	Ground Herbivore/ Omnivore	Bushy-tailed Woodrat	BTWR
		Columbian Ground Squirrel	CGSQ
		Deer Mouse	DEMO
		Golden-mantled Ground Squirrel	GMSQ
		Heather Vole	HEAV
		Hoary Marmot	HORM
		Long-tailed Vole	LTDV
		Mountain Cottontail	MTCT
		Northern Pocket Gopher	NPGO
		Pika	PIKA
		Red Squirrel	RESQ
		Southern Red-backed Vole	SRBV
		Snowshoe Hare	SNSH
		Yellow-bellied Marmot	YBMA
		Yellow pine chipmunk	YPCM
		Western Jumping Mouse	WJMO
	Aquatic Invertivore/ Omnivore	Water Shrew	WATS
		Water Vole	WATV

Weight

After the animal is secured in the plastic bag, the animal weight (minus the weight of the plastic bag) will be measured (in g) using a spring-loaded scale. Animal weight will be used to help identify the age of the organism (and possible length of contaminant exposure).

Age

In general, guidelines for aging mammals are derived from the findings of field studies that mark individuals at birth and follow them through adulthood. Aging criteria for mammals are generally taxa specific. For the most part, mammals are assigned to broad age classes (following table) relative to developmental or reproductive milestones as neonate, nestling, suckling, pouch young, juvenile, immature, subadult, adult and old adult (Kunz et al. 1996). During the Phase IIC sampling efforts, it will be difficult to assess absolute age in the field as the methods for aging rely on verifying age-related differences (e.g. body size, ossification of long bones, tooth wear) by measurement on a statistically appropriate number of known-age individuals (Kunz et al. 1996). Mammals captured in the field can be aged by collecting body measurements and evaluating reproductive criteria relative to the broad age categories listed above.

Age Categories	
Neonate	A newborn mammal with a detectable umbilical cord. Sometimes used to refer to any young animal early in lactation.
Nestling	A young animal with limited locomotory and sensory development that has not left the nest; usually young of an altricial species prior to weaning.
Suckling	A mammal before weaning.
Pouch Young	A young marsupial that has not left the pouch or, if a pouch is not present, has not detached from a teat.
Juvenile	A weaned young mammal that still associated with its mother or siblings and may nurse infrequently; usually smaller than a subadult.
Immature	A young mammal that is neither fully grown nor sexually mature.
Subadult	A young mammal that is not fully grown but that may or may not be sexually mature or have adult pelage.
Adult	A fully grown mammal that is sexually mature.
Old adult	An animal that shows extreme tooth wear and/or poor body condition.

Dead Animals

Animals found dead in a trap will be measured and checked through the plastic bag to limit exposure to ectoparasites and other health hazards associated with small mammals. Obtain side-view and belly-view photographs of the specimen. Any visible abnormalities (e.g., hair loss, presence of tumors, etc.) will be recorded on the Trap Log Form.

Mark and Release

Animals not identified for gross necropsy and the collection of tissue samples will be tagged and released. The tags will be either numbered ear tags or small marks on the underbelly with nontoxic dye or paint. If numbered ear tags are used the ear tag number will be recorded on the Trap Log Form.

4.9 Gross Necropsy and Collection of Tissue Samples

A subset of the mammals collected will be sacrificed for the examination of gross and microscopic lesions in the lungs, gastrointestinal tract, and kidney. Animals selected will be given an index ID number on the small mammal trapping log. The following targets are identified for histopathology examination:

- For each sampling location (SMT-1, -2, -3, SMT-Ref) at least 15 individuals within the ground herbivore/omnivore group will be examined
- Any shrews captured will be examined (ground invertivore exposed receptor group or aquatic invertivore/omnivore receptor group) at up to 10 individuals per sampling location)
- Similar species (within the ground herbivore/omnivore) group will be examined across sampling locations at SMT-1, -2 and SMT-ref with a goal of at least three species
- For riparian species the goal is two species
- Any arboreal invertivore collected will be examined (up to 10 individuals per sampling location)

Based on available information as previously discussed the most common species expected in the collections are the deer mouse and southern red-backed vole which are within the ground herbivore/omnivore receptor group. Pearson and Ruggiero (2003) did have some success capturing shrews using the Sherman traps with the vagrant shrew and dusky shrew being the sixth and seventh most frequently captured mammal. Shrew capture at OU3 is possible.

Live animals identified for analyses of histopathology and asbestos in tissues will be euthanized via carbon dioxide exposure. Carbon dioxide exposure is a humane method of euthanasia approved by the American Veterinary Medical Association (AVMA, 2007). Carbon dioxide will be introduced into the plastic bag containing the live animal by placing a carbon dioxide canister hose into a small section of the bag. After the animal is gassed, it will be examined to ensure death. The animal will then be placed into either a bag or plastic jar (2 to 4 oz.), depending on the species collected. The container will be labeled with a unique Index ID number on the field sampling data sheet (FSDS), as well as the time and date of collection, and stored on wet ice in a cooler until necropsy can be performed.

Samples (mammals) collected in the field and placed on wet ice will be processed as soon as possible after trap checks to reduce potential degradation of the specimens. For Libby OU3, gross necropsy and the collection of tissue samples will be performed in the field.

Necropsy results are recorded on a mammalian gross necropsy form. The necropsies will be performed by experienced and trained personnel. The general steps for necropsy include:

- When handling animals for necropsy, the primary consideration should be personal safety. Field personnel should be trained in techniques to handle mammals in a manner to minimize potential transfer of wildlife diseases. Powder free gloves should be worn at all times as well as protective clothing (e.g. protective suits, coveralls) and rubber boots.

- Depending on the size of the mammal, examination of external and internal features should be done with the unaided eye or with a dissecting microscope, whichever is appropriate.

- Examine the body surface of the mammal, and denote as normal or abnormal.

- Document presence of lesions on body surface, whether lesions are open or closed, and location of any such lesions.

- Examine the organs for color, size (swelling), and other gross abnormalities including the presence of macroscopic lesions, nodules or plaques.

- Record observations on the FSDS sheet.

All personnel within the small mammal processing area should wear disposable boot covers, disposable coveralls, and a full-face respirator equipped with a HEPA filter. During sample processing, one individual should be designated as "clean" and thus be able to assist the sample processors in packaging the specimens for shipment by performing activities such as labeling clean containers, holding containers open while samples are placed inside, and placing packaged samples in the shipping coolers. This ensures that the outer bags and coolers are not contaminated when the samples arrive at their destination.

Field personnel will complete a chain-of-custody form for collected tissue samples in accord with Libby OU3 specific SOPs including SOP#9, *Chain of Custody Procedures* and *SOP#8 Sampling Handling*.

4.10 Tissue Sample Preparation

The lung, gastrointestinal tract and kidney will be removed and portions of the tissues will be sent for histopathology examination and the analysis of asbestos levels by Transmission Electron Microscopy (TEM). A FSDS should be completed for each tissue sample processed. Sampling location, tag number, date, species, and data on the specimen metrics described below should be recorded. Procedures for tissue sample collection and preparation are described below.

- Tissue sample labels indicating the mammal index ID number assigned to the individual mammal, the type of tissue collected, the date of sample collection, and

the type of analysis specified should be affixed to all tissue collection containers for proper identification.

- Wear powder-free gloves for labeling and handling of vials, and for the dissection procedure. All wide-mouth plastic collection bottles need to be labeled and preweighed before collecting tissue samples. Ensure the balance is level with a stable zero (no sample or vial). Plastic bottles used for tissue specimens should have a wide mouth and threaded caps for secure closure. Plastic bottles eliminate the potential breakage problems. All handling of vials must be with gloved hands.

- Each tissue sample will be weighed and the weight recorded on the FSDS.

- Plastic containers should be filled with 10% buffered formalin solution at a volume of 10 times the tissue volume to ensure proper fixation. Formalin is classified as hazardous and the field team should take appropriate measures to prevent skin contact or vapor inhalation.

- Dissecting tools will be dedicated to specific procedures. Dissecting tools used to expose the internal organs will not be used to remove tissues. Dissection tools should be decontaminated prior to each use as described in Section 5.0.

- Variability between species may result in some differences in the appearance and relative size of particular organs and tissues, but their location will be similar among species.

- When dissecting tissues the field team should be careful not to squeeze or distort tissues with forceps.

- Gastrointestinal (GI) Tract. To remove the GI tract, first tie off the GI tract near the throat area to prevent content from leaking out. Cut off the esophagus above the tied-off area and gently remove the entire GI area. The GI tract should be divided into four sections: esophagus, stomach, small intestine and large intestine. The GI tract segments should be opened prior to fixation. For each GI section, the samples will be divided into two and each placed into separate wide-mouthed labeled and preweighed plastic containers each containing 10% buffered formalin solution at least 10 times the tissue volume to ensure adequate preservation.

- Liver. Remove the liver and place liver pieces into labeled, and preweighed plastic bottles containing 10% buffered formalin solution at least 10 times the tissue volume for analysis by TEM and histopathology.

- Lungs. Examine both lungs externally. If there are no obvious differences in size, shape or other gross pathology then remove each of the lungs and place in separate containers. If there are differences then each of the lungs should be

divided into two parts and each part placed into separate containers. The lungs should be removed and placed into labeled, preweighed plastic containers containing 10% buffered formalin solution at least 10 times the tissue volume for analysis by TEM and histopathology.

- Kidneys. Examine both kidneys externally. If they are symmetrical in shape and size and there are no obvious differences then place each kidney in separate containers. If there are obvious differences (size and/or shape) then place one half of each kidney into two separate containers. The kidneys should be removed and placed into labeled, preweighed plastic containers containing 10% buffered formalin solution at least 10 times the tissue volume for analysis by TEM and histopathology.

- Gross Lesions. If any lesions are noted, collect separate tissue samples for microscopic examination and other analyses. Cut a thin (1/8" – ¼") section of tissue that includes all or portions of the lesion and adjacent apparently healthy tissue. Use caution not to crush tissue in or around the lesion. Place the tissue sample in a volume of 10% buffered formalin solution in a wide-mouth plastic bottle equal to at least 10 times the tissue volume to ensure adequate preservation.

- Make certain that the containers are labeled and properly sealed to prevent leakage during transport.

- Pack the containers for shipping to minimize jarring the containers during shipment. Check with local couriers regarding current requirements or restrictions for shipment of formalin.

- Extreme temperatures can alter tissue characteristics, making tissues unsuitable for analysis. Exposure of dead specimens to extreme cold can cause tissue to freeze, making histopathological analysis difficult. Extreme heat can cause rapid decomposition of tissue. Samples should be labeled and shipped following procedures outlined in the Sample Documentation and Sample Packaging and Shipping SOPs.

4.11 Tissue Samples for Histopathology

Samples collected and preserved for histopathology should be transported to a laboratory qualified and experienced in performing histopathology examination of tissues. The histopathology laboratory will be responsible for further fixation and preparation of samples for histopathological examination.

4.12 Tissue Samples for Asbestos Residues

Samples collected and preserved for asbestos residue analyses will be transported to a Libby approved analytical laboratory.

5.0 DECONTAMINATION

To disinfect traps, at least one set of three 5-gallon buckets should be set up in the designated small mammal processing area. One bucket should be filled with dilute 5% hospital-grade Lysol™ or hypochlorite bleach solution for disinfection and the other two should be filled with tap water for rinsing. Traps should first be completely immersed in the disinfectant solution. Any visible dirt, fecal material or bait should be scrubbed off with a brush and the traps should be left to soak in the disinfectant for at least 10 minutes. After soaking, the traps should be dipped in the first and then the second bucket of rinse water, and set out to dry. When the disinfectant solution or rinse water baths become dirty with debris from the traps, the liquid should be disposed of properly, and new baths should be prepared. All waste material from small mammal activities, including used paper towels, gloves, disposable coveralls, plastic bags, etc. should be placed in a plastic garbage bag. When processing is complete, bags should be tied or taped shut and disposed of properly and all work surfaces and equipment within the small mammal processing area should be wiped down with a dilute 5% hospital-grade Lysol™ solution or a solution of 1% hypochlorite bleach.

Once sample processing is complete, personal protective clothing and equipment should be removed by first removing the outer layer of gloves, which should be discarded (if leather) or disinfected (if rubber) with a Lysol™ or hypochlorite solution. Coveralls should be removed next, followed by boot covers. The inner gloves should be washed in a disinfecting solution, washed with soap and water, and then removed and discarded. The respirator should be removed last. Personnel should then thoroughly wash bare hands with disinfectant soap and water.

6.0 QUALITY ASSURANCE/QUALITY CONTROL

All small mammal specimens shall be documented in accordance with Section 5.7 above, and chain-of-custody forms shall be completed according to SOP #9, *Chain of Custody Procedures*. A small mammal field sampling trap log (Appendix A) must be completed as well as a field sampling data sheet (FSDS) for each specimen collected, preserved and shipped for tissue burden analyses. Each sample must be kept in its own sampling jar, on which is written the index ID, initials of the field personnel collecting the sample, and collection date and time. A bound field logbook must be maintained by field personnel to record daily activities. Deviations from this sampling plan should be noted in the field notebook, as necessary. Separate entries should be made for each trap location checked.

7.0 DATA VALIDATION

All data recorded on field data sheets will be checked by the FTL against records kept in field logbooks. It is also the responsibility of the FTL to verify the contents of each shipping cooler against the chain-of-custody form prior to shipment.

8.0 HEALTH AND SAFETY

According to the Centers for Disease Control and Prevention (CDCP), several species of small mammals (e.g., *Peromyscus maniculatus, Sigmodon hispidus,* and *Microtus pennsylvanicus*) have been found to carry and potentially transmit a hantavirus to humans (CDCP 1996). Field biologists and other personnel who are exposed to small mammal body fluids and excreta are particularly at risk of hantavirus infection (Mills et al. 1995). This virus can cause hantavirus pulmonary syndrome (HPS), which has been fatal to a high percentage of exposed individuals. Individuals who plan to trap, handle, process, or otherwise be involved in any activities related to small mammals should be educated about the inherent risks of such activities, as well as ways to minimize those risks.

During summer months, small mammals may also carry external parasites such as ticks and fleas, which may transmit diseases such as Lyme disease, Rocky Mountain Spotted Fever, or Plague. Personnel should carefully inspect their clothing and wear full body tyvek when appropriate to avoid the possibility of infection by insect bites.

A limited number of people should be assigned to trap, handle and process small mammals. The sample processing area should only be entered by the personnel assigned to trap and handle small mammals. Food and drinking water is not allowed in the small mammal processing area.

When setting and checking traps, field personnel should wear surgical gloves underneath an exterior pair of leather or thick rubber gloves to prevent the interior gloves from getting torn on the sharp surfaces of the traps. Care should be taken when handling the traps to avoid injury. When checking traps and decontaminating equipment, field personnel should wear health and safety equipment as specified in the site-specific HASP. At a minimum, half-face respirators with HEPA filters should be worn. In dry or dusty conditions, disposable coveralls (e.g., tyvek) and appropriate eye protection.

During processing of small mammals in the field, full face respirators fitted with HEPA filters (or half face respirators along with appropriate eye protection) should be worn, along with two layers of chemical resistant surgical gloves or one layer of surgical gloves and one layer of thick nitrile gloves.

9.0 REFERENCES

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FIGURES

Figure 1
Sherman Live Trap

APPENDIX A
SMALL MAMMAL TRAPPING LOG

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LIBBY OU3 PHASE IIC

Small Mammal Trapping Log

Sampling Location ID: ______________________________

Trap Set Date and Time	Trap Check Date and Time	Trap ID	Species Collected	Weight (g)	Alive (A)/Dead (D)	Age	Notes of Physical Abnormalities	Photo Number	Index ID

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LIBBY OU3 PHASE IIC

Small Mammal and Avian Sampling Location Information

Sampling Location ID	Description	GPS Units	xCoor	y Coor	Photo Number

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Date: June 9, 2008 SOP BIRD-LIBBY-OU3 (Rev. 0)

Title: SAMPLING AND ANALYSIS OF BIRDS FOR ASBESTOS

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	_____ Bonita Lavelle, USEPA RPM	_____
SOP Author	_____ Janet Burris, SRC	_____

Revision Number	Date	Reason for Revision
0	05/28/2008	--

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for collection of birds using mist nets for biological surveys, chemical analysis of tissues and/or histopathological examination. This procedure will be used by USEPA Region 8 for the Remedial Investigation work for Operable Unit 3 performed at the Libby Asbestos Superfund site.

This document focuses on methods and equipment that are readily available and typically applied in collecting avian samples. It is not intended to provide an all-inclusive discussion of bird collection methods. Specific sampling problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be clearly described in the project-specific sampling plan and approved by the Project Manager and the Quality Manager.

2.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects site-specific sampling methods, sample locations, and constituents to be analyzed with input from other key project staff.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Biologist: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL, field biologist, or engineer in the implementation of tasks. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

3. 0 EQUIPMENT

3.1 Mist Netting

- State and federal permits (recommended, but not required for samples collected under Superfund)

- Equipment for clearing brush (bank blades, machetes, etc.)
- Mist nets and poles
- Pole pounder (sledge hammer and bolt)
- Holding bags
- Pesola scale
- GPS unit
- Identification guides
- Supply box
- Crochet hook
- Field sample data sheet(s) for bird collection
- Field log book
- Ink pen
- Digital camera

3.2 Tissue Collection

- Powder free gloves
- Wide-mouthed plastic bottles of varying sizes with lids
- Container labels
- Scale
- Ethanol
- Alconox
- Reagent-grade nitric acid
- Squirt bottles
- Deionized water
- Scalpel
- Stainless dissecting forceps
- Stainless dissecting scissors
- Dissecting tray
- Work lights (head or table mounted)
- Permanent black ink pens
- Field sample data sheet(s) for tissue collection
- Field log book
- Paper towels
- Kimwipes

4.0 SAMPLING PROCEDURES

4.1 Preparation

A scientific collection permit should be obtained from the appropriate federal or state agency. Most states have permit information available on the internet. A natural heritage search for threatened or endangered species should also be requested from the state. In addition, permission from the landowner(s) must be received prior to trapping at the site or reference areas.

4.2 Sampling Locations

Four areas are identified for the collection of birds. These locations are listed in the following table along with the rationale for their selection. The exact locations of the sampling areas and placement of trap lines will be made during an initial field reconnaissance. Sampling locations will be selected based on the identified habitats, terrain, access and other considerations.

Location ID (Same area as)*	General Descriptions and Rationale	General Identified Areas
MN-1 (SMT-1)	Area on the Mine Site Disturbed Area. This area is expected to have highest the highest asbestos exposures but not the best habitat to support species.	MW-6 or MW-16
MN-2 (SMT-2)	Near the disturbed Mine Site Area in an area with better habitat than SMT-1 (meadow/forested) with known asbestos contamination in soils, tree bark and duff.	Near SL-45-01
MN-3 (SMT-3)	Riparian area near water body with both established use by waterfowl and/or shorebirds and known asbestos contamination in sediments and/or surface water.	Tailings Pond
MN-Ref (SMT-Ref)	Reference area with habitat matched closely in terms of vegetative cover and elevation to SMT-2.	Area upwind of OU3 to the west

*Small Mammal Trap (SMT) locations in SOP MAMMAL-LIBBY-OU3 (Rev.0)

4.3 Targeted Species

The targeted avian species for collection in the mined area and forested area the ground foraging species (invertivore and omnivore). The targeted species in the riparian area are aquatic herbivores/omnivores. The following table provides the list of mammalian wildlife Exposed Receptor Groups for Libby OU3 and species within each group.

Exposed Receptor Group		Description of Exposed Group	Species in Group Common name *(Genus species)*-Number Reported[1]
Avian	Ground Invertivore	Avian insectivorous species that feed primarily on soil invertebrates.	American robin (*Turdus migratorius*) - 828 Common Yellowthroat (*Geothlypis trichas*) 37 Flammulated Owl (*Otus flammeolus*) - 32 House Wren (*Troglodytes aedon*) - 16 Killdeer (*Charadrius vociferus*) - 19 Nashville Warbler (*Vermivora ruficapilla*) -58 Northern Flicker (*Colaptes auratus*) -575 Rock Wren (Salpinctes obsoletus) - 11 Spotted Towhee (*Pipilo maculatus*) - 78 Townsend's Solitaire (*Myadestes townsendi*) - 515 Warbling Vireo (*Vireo gilvus*) - 435 Winter Wren (*Troglodytes troglodytes*) - 487 Western Bluebird (*Sialia mexicana*) - 11 American Redstart (*Setophaga ruticilla*)

Exposed Receptor Group	Description of Exposed Group	Species in Group Common name *(Genus species)*-Number Reported[1]
Arboreal Invertivore	Avian species that feed primarily in trees on invertebrates.	American Three-toed Woodpecker (*Picoides dorsalis*) Black-backed Woodpecker (*Picoides arcticus*) Black-capped Chickadee (*Poecile atricapillus*) Brown Creeper (*Certhia Americana*) Chestnut-backed Chickadee (*Poecile rufescens*) Downy Woodpecker (*Picoides pubescens*) Golden-crowned Kinglet (*Regulus satrapa*) Orange-crowned Warbler (*Vermivora celata*) Pileated Woodpecker (*Dryocopus pileatus*) Pygmy Nuthatch (*Sitta pygmaea*) Red-breasted Nuthatch (*Sitta canadensis*) Ruby-crowned Kinglet (*Regulus calendula*) Townsend's Warbler (*Dendroica townsendi*)
Ground Herbivore	Avian species that feed primarily on plant material and forage on the ground.	Chipping Sparrow (*Spizella passerina*) - 969 Common Redpoll (*Carduelis flammea*) - 3 Pine Siskin (*Carduelis pinus*) - 1213 Spruce Grouse (*Falcipennis canadensis*) - 16 Mourning Dove (*Zenaida macroura*) - 24 Ruffed Grouse (*Bonasa umbellus*) - 148
Aquatic Invertivore	Avian species that forage in along streams and ponds probing into sediments.	American Dipper (*Cinclus mexicanus*) - 20 Bank Swallow (*Riparia riparia*) - 8 Bufflehead (Bucephala albeola) - 5 Marsh Wren (*Cistothorus palustris) - 7* Rufous Hummingbird (*Selasphorus rufus*) - 49 Spotted Sandpiper (*Actitis macularius*) - 29
Aquatic Herbivore/ Omnivore	Avian species that feed on aquatic vegetation and sometimes aquatic invertebrates	American Coot (*Fulica americana*) - 9 American Wigeon (*Anas americana*) - 5 Blue-winged Teal (*Anas discors*) - 6 Green-winged Teal (*Anas crecca*) - 6 Mallard (*Anas platyrhynchos*) - 34

[1]Number of occurrences in Lincoln, County Montana (http://fieldguide.mt.gov/)
If number is not presented, the data were not available.

Based on the number of recorded sighting of species within these groups within Lincoln County in the Montana Natural Heritage Program Animal Tracker (http://fieldguide.mt.gov/), the species are expected to be the most commonly collected include the American robin (*Turdus migratorius*), the Northern flicker (*Colaptes auratus*), Townsend's Solitaire (*Myadestes townsendi*), warbling vireo (*Vireo gilvus*), winter wren (*Troglodytes troglodytes*), chipping sparrow (Sizella passerine), pine siskin (*Carduelis pinus*) and ruffed grouse (*Bonasa umbellus*). For riparian species the most common species include the mallard (*Anas platyrhynchos*) and spotted sandpiper (*Actiis macularius*).

4.4 Sampling Method

This SOP is focused on the use of mist nets to capture birds. The use of mist nets for monitoring bird populations is reviewed by Ralph and Dunn (2004). Mist netting is often used to identify what species are present within a collection area as well as abundance. Mist nets are fine mesh

nets of differing gauge and length. The nets are made of thin "invisible" threads (1" squares) and are mounted on wooden or aluminum poles at differing heights above the ground (Figure 1).

Most mist netting programs are focused on the collection of birds for diversity and abundance information. For these studies, the method is less biased compared to census methods (visual and/or auditory surveys) as it is not affected by the observer's skills at recognizing birds visually and/or their auditory calls. The method also allows for the physical collection of birds for further examination and banding (histopathology and tissue residues of contaminants). The method collects more ground-foraging and non-singing birds compared to auditory and visual surveys and misses some species such as aerial insectivores and raptors (Ralph and Dunn, 2004). Birds that spend their time in the canopy (20-30 m) will rarely be caught in a 2 m high mist net, whereas birds that spend all of their time within 2 m of the ground are likely to be caught frequently (Remsen and Good, 1996).

Mist netting was selected for use at Libby OU3 based on the goals of the program to collect ground foraging birds for the collection of tissues and the examination of histopathology and asbestos residues. The collection of information on avian diversity and abundance would require a much higher level of effort and more complex design. For example, the Monitoring Avian Productivity and Survivorship (MAPS) which is a cooperative effort among public agencies, private organizations, and individual bird ringers in North America that operates a network of over 500 constant-effort mist netting stations, recommends operating nets one day per 10-day period during the breeding season (May to August) (MAPS 2008).

The recommended procedures for mist net type and set up (Section 4.4), number and placement (Section 4.5) and monitoring (Section 4.6) are based on a review of Ralph and Dunn (2004) as well as an available protocol from the U.S. Fish and Wildlife Service Upper Columbia Fish and Wildlife Office in Spokane Washington and selected other publications.

4.4 Mist Net Type and Set Up

For the Phase IIC sampling at Libby OU3 the following guidelines for mist net set up shall be followed:

- The mist net should be 12 m long, 2.6 m high with 30 - 36 mm mesh with four tiers and black tethered nylon net. The nets should be erected with the bottom of the lower shelf touching the ground.

- The mist net poles should be made of galvanized steel or aluminum conduit. Each pole should consist of one 5 foot section of ¾" diameter conduit and one 5 foot section of ½" diameter conduit.

- To set up the net, the large pole should be pounded into the ground between 4-6 inches deep by placing a large bolt into the top of the conduit and pounding on the bolt with a sledgehammer. Once the base is firmly in the ground, place the end loops of the mist net over the base. The smaller pole should be inserted into the top of the larger pole to provide 8 to 9 feet of pole height to mount each end of the net. The extent of overlap between the

poles should be minimized to approximately 6 inches. To restrict the extent of overlap, a small hole should be drilled in the lower pole and a small bolt inserted through the hole prior to inserting the top pole. Spread the mist net loops evenly across the pole. Unfurl the net and stretch it tight across the area where the net will be positioned. Mark the spot where the net stretches to and pound another pole base into the ground. Place the end loops of the mist net over the pole base, and insert the top portion of the pole. Spread the mist net evenly up and down both poles.

4.5 Mist Net Placement and Operation

For the Phase IIC sampling at Libby OU3 the following guidelines for mist net placement shall be followed:

- The nets should be placed against a dark background (usually dense vegetation) so that they will not be visible to the birds.
- Photographs of the mist net and surrounding habitat will be recorded.
- Data on the habitat within the area will be recorded on the Avian Sampling Location Log form.
- Five mist nets will be placed per sampling area set at 20 to 80 m apart.
- Locations of individual mist nets within the sampling area will be recorded using a hand-held Global Positioning System (GPS) unit. The locations will also be sketched on the Avian Sampling Location Log for or field log book.
- If nests of the target species are found, it will be acceptable to place nets such that the nesting adults are captured when leaving or coming to the nest.
- Nets will be monitored from within 15 minutes of dawn and operated for five hours, weather permitting. Nets will not be operated during times of rain.
- Nets should be set in an area where access and retrieval of birds is easy.
- Nets will only be operated at one location within a sampling area on one day only. Several studies (Ralph and Dunn, 2004; MacArthur and MacArthur, 1974; Remsen and Good, 1996) suggest that operation of nets over consecutive days at the same location is not effective in capturing more birds. Birds quickly learn to avoid the nets.

4.6 Net Monitoring and Collection of Birds

For the Phase IIC sampling at Libby OU3 the following guidelines for mist monitoring shall be followed:

- Once nets are set they are checked regularly, at least once every 30 minutes. Nets should not be set when ambient temperatures are below 0º C or above 27º C (80.5 Farenheight).

- No activity should take place near nets between net checks, and all captures should be passive (no chasing or tape lures).

Data on the birds collected from each net will be recorded on the Avian Capture Log (Appendix A). The birds collected will be labeled and numbered as follows:

  - MN-__-x-y
  - MN-__ equals the sampling location number
  - X is the unique net number (1 through 5)
  - Y is the unique bird identification number

Removing birds from mist nets should be done with caution. The following guidelines shall be followed:

- Care should be taken when removing birds from the net so as not to subject them to injury and minimize stress. To remove a bird from the net, the field team should attempt to determine from which direction the bird entered the net and remove it from that side. Since birds generally fly into the net headfirst, usually the best method for removing the bird from the net is in the reverse order: tail, feet, wings and the head. A crochet hook may be useful in removing the net from around the bird.

- A subsample of birds will be sacrificed for the examination of gross pathology (internal and external) and the collection of tissues for histopathology and the measurement of asbestos residues. This is described in Section 5.0. Birds targeted for further analyses should be placed in a cloth holding bag.

- Live birds not targeted for further analysis should be released as soon as possible. Clip one right side tail feather approximately one half of the shaft length to clearly identify that the bird has previously been captured. This will allow for the analysis of recapture of birds.

4.7 Measurements

After removing the bird from the net, data on species and condition of the bird should be recorded on the Bird Collection Log form. When possible, age and sex (breeding status) should also be noted. All members of the field team should be trained in techniques used to handle birds and to identify bird species, age and sex, as well as make external examinations.

Photographs

All birds collected will be photographed and the photograph number recorded on the Bird Collection Log form.

Species

The species is recorded by a common name abbreviation according to a standard list of four-letter (English name) alpha codes compiled for the Institute for Bird Populations by Pyle and DeSante (www.birdpop.org). The abbreviations for the target species at Libby OU3 are listed in the following table.

Exposed Receptor Group	Common name and Code	
Ground Invertivore	American robin	AMRO
	Common Yellowthroat	COYE
	Flammulated Owl	FLOW
	House Wren	HOWR
	Killdeer	KILL
	Nashville Warbler	NAWA
	Northern Flicker	NOFL
	Rock Wren	ROWR
	American Redstart	AMRE
	Spotted Towhee	SPTO
	Townsend's Solitaire	TOSO
	Warbling Vireo	WAVI
	Western Bluebird	WEBL
	Winter Wren	WIWR
Arboreal Invertivore	American Three-toed Woodpecker	ATTW
	Black-backed Woodpecker	BBWO
	Black-capped Chickadee	BCCH
	Brown Creeper	BRCR
	Chestnut-backed Chickadee	CBCH
	Downy Woodpecker	DOWO
	Golden-crowned Kinglet	GCKI
	Orange-crowned Warbler	OCWA
	Pileated Woodpecker	PIWO
	Pygmy Nuthatch	PYNU
	Red-breasted Nuthatch	RBNU
	Ruby-crowned Kinglet	RCKI
	Townsend's Warbler	TOWA
Ground Herbivore	Chipping Sparrow	CHSP
	Common Redpoll	CORE
	Pine Siskin	PISI
	Mourning Dove	MODO
	Ruffed Grouse	RUGR
	Spruce Grouse	SPGR
Aquatic Invertivore	American Dipper	AMDI
	Bank Swallow	BANS
	Bufflehead	BUFF
	Marsh Wren	MAWR
	Rufous Hummingbird	RTAH
	Spotted Sandpiper	SPSA
Aquatic Herbivore/ Omnivore	American Coot	AMCO
	American Wigeon	AMWI
	Blue-winged Teal	BWTE
	Green-winged Teal	GWTE

Exposed Receptor Group	Common name and Code	
	Mallard	MALL

Sex

Information on the sex should be recorded for each bird where possible. Techniques for determining the sex of a bird is dependent upon species. In general, birds may be sexed based on variation in plumage, size or behavior. Some species of birds can be sexed based on reproductive structures such as a brood patch in females or a cloacal protuberance in males. If sex can be determined it should be recorded on the Bird Collection Log Form. Sexing guidelines for some species can be found online at http://www.migrationresearch.org/mbo/id/idlibrary.html.

Age

There is no standard method for aging birds collected in the field. Guidelines for aging birds come from findings of banding studies that have followed banded birds from nestlings to adults. For the most part, the wing provides the most information about age based on molting of the primary and secondary coverts. A bird can be classified as juvenile (juv), hatchling year (HY), after hatchling year (AHY), second year (SY), after second year (ASY), and in some cases, third year (TY) and after third year (ATY). As part of the Libby OU3 sampling effort for analyzing asbestos in bird tissues, it is important to collect older birds for tissue analysis to gain a better understanding of the effects following potential long term exposure to asbestos particles. Unfortunately, most bird species cannot be reliably aged beyond after-second-year. However, most birds demonstrate slow aging rates and long life spans relative to their body size (Holmes and Ottinger, 2005) indicating that adult birds captured during Phase II sampling efforts will most likely range in ages of at least three to five years up to potentially 10 or more years of age depending on the species (Holmes and Ottinger, 2005; USGS 2007). If age can be determined it should be recorded on the Bird Collection Log Form. Aging guidelines for some species can be found online at http://www.migrationresearch.org/mbo/id/idlibrary.html.

Gross External Pathology

All birds collected will be examined for any external signs of abnormalities and/or parasites. The following may be included in the gross external examination: examination of body surface and denote as normal or abnormal. The bird should be examined for general appearance, feathering, pigmentation of skin and shanks, physical injuries, facial tissues, eyes, feces, nasal and respiratory discharges, respiration, gait, leg/join deformities and external parasites (Butcher and Miles 1993)

5.0 SAMPLE PREPARATION AND ANALYSIS

A subset of the birds collected will be sacrificed for the examination of gross and microscopic lesions in the lungs, air sac, gastrointestinal tract, and kidney. The following targets are identified for histopathology examination:

- For each sampling location (MN-1, -2, -3, MN-Ref) at least 15 individuals within the ground invertivore and herbivore exposed receptor groups will be examined
- Similar ground invertivore and herbivore species will be examined across sampling locations with the goal of at least three species.
- For the riparian area, up to 10 individuals will be examined representing at least two species
- Any arboreal invertivore collected will be examined (up to10 individuals per sampling location)

The following sections describe analyses of biological parameters, tissue contaminant analyses, and histopathology.

5.1 Measuring bird body weights

Due to stress to captured birds, only birds targeted for tissue analysis will be weighed. The bird can be weighed using a Pesola scale. The bird should be placed into a cloth holding bag. Body weight can be collected away from the bird capture location. The bag used to the hold the bird for weighing should be tared from the scale weight prior to placing the bird into the bag. All members of the processing staff should be trained in techniques to handle birds and make weight measurements. Inconsistencies in the way these measurements are taken can lead to errors.

5.2 Gross Bird Necropsy

A gross necropsy may be completed in the field in birds sacrificed for further external and/or internal examination and/or the collection of tissue samples (described in Section 6.3). These procedures are based on the U.S. Fish and Wildlife Service SOP 1019.3766, and U.S. Geological Society's (USGS) Field Manual of Wildlife Diseases (USGS, 1999). The general steps for bird necropsy include:

- When handling birds for necropsy, the primary consideration should be personal safety. Field personnel should be trained in techniques to handle birds in a manner to minimize potential transfer of wildlife diseases. Powder free gloves should be worn at all times as well as protective clothing (e.g. protective suits, coveralls) and rubber boots.

- Birds should be euthanized with a chemical anesthetic before being examined.

- Depending on the size of the bird, examination of external and internal features should be done with the unaided eye or with a dissecting microscope, whichever is appropriate.

- Examine the body surface of the bird, and denote as normal or abnormal.

- Document presence of lesions on body surface, whether lesions are open or closed, and location of any such lesions.

- Birds are dissected if specific tissues and/or organs are being collected for histopathology or for contaminant residue analysis. Begin the dissection by placing the dead bird on its back in a dissecting tray. Gently blow on the breast of the bird to expose the unfeathered area along the sternum. Spray ethanol on this area to dampen the feathers and sterilize the area. Cut the skin from the posterior region of the sternum to the abdomen with a scalpel. Be careful not to penetrate the body cavity, particularly the abdominal region. Continue the skin incision to the vent at the base of the bill. Use your fingers to reflect the skin away from the next, breast, and abdominal areas to expose the breast muscle. Cut the musculature with dissecting scissors at the edge of the breast muscles. Insert the thumb of one gloved hand into the incision along the midpoint of the sternum and apply a slight pressure upwards. With dissecting scissors in the other hand, carefully cut through the ribs extending the cut on each side of the breast through the area of the wishbone. Gently separate the breastplate from the carcass; use scissors to sever any connections and push aside the air sacs.

- Examine the organs for color, size (swelling), and other gross abnormalities including the presence of macroscopic lesions, nodules or plaques.

- Record observations on the FSDS sheet.

5.3 Tissue Sample Preparation

Portions of the tissues collected from specific organs (lungs, air sac, gastrointestinal tract and kidney) will be sent for analysis of asbestos levels by Transmission Electron Microscopy (TEM), and the remaining portions will be sent for histopathological examination. A FSDS should be completed for each tissue sample processed. Sampling location, tag number, date, species, and data on the specimen metrics described below should be recorded. A gross necropsy should be performed as described in Section 6.2 for each bird prior to collection of tissue samples. Procedures for tissue sample collection and preparation are described below.

- Tissue sample labels indicating the station from which the bird was collected, the sample number assigned to the bird, the type of tissue collected, the date of sample collection, and the type of analysis specified should be affixed to all tissue collection containers for proper identification.

- Wear powder-free gloves for labeling and handling of vials, and for the dissection procedure. All wide-mouth plastic collection bottles need to be labeled and preweighed before collecting tissue samples. Ensure the balance is level with a stable zero (no sample or vial). Plastic bottles used for tissue specimens should have a wide mouth and threaded caps for secure closure. Plastic bottles eliminate the potential breakage problems. All handling of vials must be with gloved hands.

- Plastic containers should be filled with 10% buffered formalin solution at a volume of 10 times the tissue volume to ensure proper fixation. Formalin is classified as hazardous and the field team should take appropriate measures to prevent skin contact or vapor inhalation.

- Dissecting tools will be dedicated to specific procedures. Dissecting tools used to expose the internal organs will not be used to remove tissues. Dissection tools should be decontaminated prior to each use as described in Section 6.4.

- The histopathology laboratory selected will be consulted as to the size of the tissues submitted for histopathology. When collecting tissues, the field team should place one half of the tissue in a container for histopathology and one half in a container for asbestos tissue residue analyses.

- Species variation may results in some differences in the appearance and relative size of particular organs and tissues, but their location will be similar among species.

- When dissecting tissues the field team should be careful not to squeeze or distort tissues with forceps.

- **Gastrointestinal Tract**. To remove the GI tract, first tie off the GI tract near the throat area to prevent content from leaking out. Cut off the esophagus above the tied-off area and gently remove the entire GI area. The GI tract should be opened prior to fixation. Segments set aside for histopathology are best if ½" to 1" long (Butcher and Miles 1993). Tissue samples should be placed into wide-mouthed labeled and preweighed plastic containers each containing 10% buffered formalin solution at least 10 times the tissue volume to ensure adequate preservation.

- **Lungs.** The lungs of birds are relatively small organs. Look for two flattened structures pressed against the ribs and lying on either side of the vertebral column. The lungs should be removed and placed into labeled, preweighed plastic containers containing 10% buffered formalin solution at least 10 times the tissue volume for analysis by TEM and histopathology.

- **Air sacs.** The air sacs extend out from the lungs. There should be a pair of abdominal, two pairs of thoracic, one pair of subscapular, one axillary pair, and one cervical pair. The sacs squeeze into any available space between the other internal organs. If possible, dissect out the air sacs and place them into labeled, preweighed plastic containers containing 10% buffered formalin for analysis by TEM and histopathology. It may be difficult to excise the air sacs, as most will be deflated.

- **Kidneys.** Along the dorsal wall of the body cavity lie the large, lobed kidneys of the urinary tract. The kidneys should be removed and placed into labeled, preweighed plastic containers containing 10% buffered formalin solution at least 10 times the tissue volume for analysis by TEM and histopathology.

- **Gross Lesions.** If any lesions are noted, collect separate tissues samples for microscopic examination and other analyses. Cut a thin (1/8" – ¼") section of tissue that includes all or portions of the lesion and adjacent apparently healthy tissue. Use caution not to crush tissue in or around the lesion. Place the tissue sample in a volume of 10% buffered

formalin solution in a wide-mouth plastic bottle equal to at least 10 times the tissue volume to ensure adequate preservation.

- Make certain that the containers are labeled and properly sealed to prevent leakage during transport.

- Pack the containers for shipping to minimize jarring the containers during shipment. Check with local couriers regarding current requirements or restrictions for shipment of formalin.

- Extreme temperatures can alter tissue characteristics, making tissues unsuitable for analysis. Exposure of dead specimens to extreme cold can cause tissue to freeze, making histopathological analysis difficult. Extreme heat can cause rapid decomposition of tissue. Samples should be labeled and shipped following procedures outlined in the Sample Documentation and Sample Packaging and Shipping SOPs.

5.4 Equipment Decontamination

If dedicated sample equipment is not used, prior to each use with another bird, all dissecting tools and trays contacting bird tissue must be cleaned. The solutions for cleaning are based on the cleaning procedure described by the U.S. Fish and Wildlife Service in SOP 1019.3766. These solutions include a weak alkanox soap solution, 5% nitric acid solution, and deionized water, all in squirt bottles. The weak alkanox soap solution is sprayed on the tool and the edges of the tool are gently scrubbed with a kimwipe. The tool is sprayed with deionized water to wash soap off. The tool is rinsed with nitric acid solution, then rinsed with deionized water and placed back onto a clean kimwipe at the dissecting area.

The alkanox solution should be made in one liter increments by placing approximately one gram of alkanox in the prelabeled bottle, and diluting with tap water. The nitric acid solution is also made in one liter increments by filling the bottle 50% full with deionized water, adding 50 ml of reagent-grade concentrated nitric acid, then filling the bottle to the neck with deionized water. DO NOT add acid first, a violent reaction will results when the deionized water is added. Any spent wipes, paper towels, or other decontamination waste materials must be disposed or stored properly as investigation-derived waste.

5.5 Histopathology

Samples collected and preserved for histopathology should be transported to a laboratory qualified and experienced in performing hispathology examination of tissues. The histopathology laboratory will be responsible for further fixation and preparation of samples for histopathological examination.

5.6 Tissue Residue

Samples collected and preserved for asbestos residue analyses will be transported to a Libby approved analytical laboratory.

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

All specimens shall be documented in a chain-of-custody form shall be completed according to SOP #9, *Chain of Custody Procedures*. A bird collection log (Appendix A) will be completed as well as a field sampling data sheet (FSDS) for each specimen collected, preserved and shipped for either histopathology or tissue burden analyses. Each sample must be kept in its own sampling jar, on which is written the index ID, initials of the field personnel collecting the sample, and collection date and time. A bound field logbook must be maintained by field personnel to record daily activities. Deviations from this sampling plan should be noted in the field notebook, as necessary. Separate entries should be made for each trap location checked.

7.0 DECONTAMINATION AND HEALTH AND SAFETY

All equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

All field crew members will conduct sampling in accordance with the appropriate level of health and safety training required by their parent organization.

8. 0 REFERENCES

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Figure 1. Photos of Mist Nets

Date: February 7, 2008

SOP DUFF-LIBBY-OU3 (Rev. 0)

Title: SAMPLING AND ANALYSIS OF DUFF FOR ASBESTOS

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	Mary Goddard for Bonnie Project Chemist Bonita Lavelle, USEPA RPM	2/7/08
SOP Author	W J Brattin William Brattin, SRC	2/7/08

Revision No.	Date	Reason for Revision
0	02/07/2008	--

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for collection and analysis of duff samples for Libby amphibole asbestos (LA). Duff consists of the un-decomposed twigs, needles and other vegetation and the layer of partially- to fully-decomposed litter that occurs on top of the mineral soil in forested areas. This procedure will be used by USEPA Region 8 for the Remedial Investigation work for Operable Unit 3 performed at the Libby Asbestos Superfund site.

2.0 RESPONSIBILITIES

The Field Sampling Team Leader is responsible for ensuring that all duff samples are collected in accord with this SOP. The Laboratory Director is responsible for ensuring that duff samples provided to the laboratory for evaluation by this SOP are prepared and analyzed in accord with the requirements of this SOP. It is the responsibility of the Field Sampling Team Leader and the Laboratory Director to communicate the need for any deviations from the SOP with the appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist.

3. 0 EQUIPMENT

3.1 Field Equipment

- Ziploc® plastic bags
- sample identification labels
- GPS unit
- field log book
- field sample data sheet(s)
- ink pen
- clear packaging tape

3.2 Laboratory Equipment /Reagents

- Large aluminum trays
- Drying oven
- Large metal tray(s) (large enough for duff sample to cover bottom up to 1/2 in.)
- Muffle furnace
- Glass stirring rods
- Fume hood
- HEPA filtered hood
- Reagent grade or better acetone
- Reagent grade or better HCl
- Fiber-free deionized water (FDI water)
- Ultrasonic bath, producing a rate of energy deposition in the range of 0.08-0.12 MW/m^3
- Disposable plastic filter funnel apparatus

- Disposable filter funnels with straight sides [VWR # 145-0020]
- Culture dishes [VWR # 25388-581, case of 500]
- 47 mm 0.45 micron MCE or 0.4 micron PC filters
- Kim wipes or alternative paper
- Ziploc plastic bags
- Glass petri dishes
- Glass microscope slides
- Low temperature plasma asher
- Vacuum evaporator (carbon coater)
- Graphite or carbon rods
- HEPA laminar flow hood
- Acetone vapor generator
- Grids
- Fine forceps
- Grid storage boxes
- Jaffe wick or sponge
- Transmission electron microscope with the following capabilities:
  - 100 Kev
  - fine probe size <250 nm
  - Energy Dispersive X-Ray Analysis (EDXA)
  - Selective Area Electron Diffraction (SAED)

4.0 METHOD SUMMARY

A duff sample is collected by hand at a selected field location and placed in a plastic bag. Duff samples are prepared for analysis by high temperature ashing to remove organic matter. The residue is then analyzed for LA by transmission electron microscopy (TEM) and/or by Polarized Light Microscopy (PLM), as specified in the project-specific Sampling and Analysis Plan (SAP).

5.0 SAMPLE COLLECTION

Duff samples should be collected from the soil sampling stations specified in the project-specific SAP. At each specified sampling station, collect any fresh or partially decayed organic debris (e.g., twigs, leaves, pine needles) using a freshly-gloved hand from the soil surface within an area that is approximately 6 in. x 6 in. Care should be taken to ensure that the top layer of soil beneath the organic debris is not included in the duff material sample. Place the duff material into a large, air-tight, re-sealable plastic bag. Label the bag with the same sample identifier as the soil field sample, and place clear packaging tape over the sample identifier label.

Attachment A provides a Field Sample Data Sheet (FSDS) for recording field information on each duff sample. [Note: in some cases, an alternative FSDS may be specified and provided in the project specific SAP]. Note any special circumstances or conditions about the sampling location. Obtain and record the GPS coordinates of the sampling location on the FSDS form.

6.0 SAMPLE PREPARATION AND ANALYSIS

6.1 Drying and Ashing

Weigh and record the tare weight of a clean, dry aluminum tray of approximately quart size. Fill the aluminum tray to approximately ¾ full. The samples may be split across as many trays as may be needed, providing the samples' identification number is clearly marked on each tray. In addition, for tracking purposes each tray should possess a mark to make it unique and identifiable from the other trays. This identifier shall be recorded in the laboratory preparation logs. Each tray will need to be initially tared and then gravimetrically tracked through the process. Place the tray(s) with the sample into a drying oven. Heat to 60°C and hold at this temperature until weight stabilizes (at least 10 hours). Record the dry weight and calculate the mass of the dried duff sample by the difference.

Once samples are dried, they then shall be ashed. Weigh and record the tare weight of one or more clean metal pans capable of withstanding the heat of a 450°C oven. Working under a hood, transfer the dried duff to the tared pan(s), place a lid on the pan and move to a muffle furnace. Ramp up the furnace from a cold start to 450°C and hold at this temperature for 18 hours or until all organic matter is removed.

Allow the pan(s) to cool. Remove the lid(s), weigh and record the mass of the pan(s) plus the ashed residue. Calculate the mass of the ashed residue in each pan by difference. If the sample was ashed in more than one pan, compute the total mass of the ashed residue for the sample by summation across pans.

Under a laminar flow hood, slowly pour the ash from each sample into a Ziploc bag. If the sample was ashed in more than one pan, all the pans for that sample are combined into a single Ziploc bag. If the ash still retains some structure, seal the bag tightly and manipulate the ash by hand to reduce it to a fine homogenous powder. Invert the bag 3-4 times to thoroughly mix the ash.

All information regarding sample preparation shall be recorded using the sample preparation log sheet, presented as Attachment B.

6.2 TEM Analysis

Acid Treatment

Remove an aliquot of approximately 0.25 g of the well-mixed ash and place into a crucible. Record the weight (measured to an accuracy of ± 0.01 g) on the sample preparation data sheet (see Attachment B). To the ashed residue in the crucible, add just enough FDI water (approx 1-2 mL) to cover the surface of the residue. Slowly add concentrated HCl to the wetted ash (approx. 10-20 mL). Typically a visible effervescing is observed. Add the HCl slowly to keep this reaction controlled. A small glass stirring rod is useful at this point to gently stir the ash and expose all material to the acid.

If after 3-5 minutes there is no further visible reaction, proceed to the next step. If bubbling is still occurring, continue observation and gentle stirring for up to an additional 5 minutes.

Dilute the sample by adding FDI water directly to the crucible (approx 20 mL) using a squirt bottle. Pour the sample into an unused disposable 100 mL specimen container with lid. Rinse out any remaining residue from the crucible into the specimen container. Do not exceed 100 mL total volume. Bring the total volume to 100 mL with DI water.

Cap the specimen cup and agitate the sample by inversion 5 or 6 times. Loosen the cap slightly and sonicate for 2 minutes. After sonication, tighten the cap and then dry the exterior of the specimen container with a laboratory wipe.

Filtration

Agitate the sample by inversion 5 or 6 times. Withdraw an initial aliquot of 0.1 to 1 mL of sonicated sample. Transfer this aliquot into a new disposable specimen container with lid. Bring the volume up to approximately 100 mL with FDI water. Cap and agitate by inversion (5 or 6 times).

Filter this entire volume onto a 47 mm mixed cellulose ester (MCE) filter with 0.4 um pore size.

If the filter appears overloaded (overall particulate level > 20%), repeat the process above, selecting a smaller aliquot volume, as suggested by the degree of overloading. Conversely, if the filter looks too lightly loaded, filter a larger aliquot.

After filtration, transfer the filter membranes to individual disposable labeled Petri dishes with lids. With Petri dish covers ajar, gently air dry the filters in a HEPA protected environment.

TEM Examination

Prepare 3 grids for TEM analysis as detailed in International Organization for Standardization (ISO) TEM method 10312, also known as ISO 10312:1995(E). Utilize 2 grids for analysis, holding the third in case of problems. After analysis, archive all three grids for potential future reanalysis.

Counting rules

Examine the grids using TEM in accord with ISO 10312 and all relevant Libby site-specific modifications, including the most recent version of LB-000016, LB-000019, LB-000028, LB-000029, LB-000029a, LB-000030, LB-000053, and LB-000066. All fibrous amphibole structures that have appropriate Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra, and having length greater than or equal to 0.5 um and an aspect ratio (length: width) $\geq$ 3:1, will be recorded on the Libby site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheet for TEM analysis of duff samples. Data recording for chrysotile (if observed) is not required.

Stopping rules

The target analytical sensitivity for sample analysis should be specified in the SAP. In the absence of a project-specific target sensitivity, the default sensitivity should be 1E+07 $(grams)^{-1}$, which is likely to correspond to a mass fraction of less than about 0.005 grams asbestos per gram duff (dry wt). The analytical sensitivity is calculated using the following equation:

$$S = \frac{EFA}{GO \cdot Ago \cdot Mass \cdot F}$$

where:

S	=	Sensitivity (1/g dry wt)
EFA	=	Effective filter area (mm^2)
GO	=	Number of grid openings counted
Ago	=	Area of one grid opening (mm^2)
Mass	=	Mass of the dried (but not ashed) duff sample (g)
F	=	Fraction of the starting duff sample applied to the filter

Count the sample until one of the following occurs:

- The target sensitivity is achieved.
- A total of 50 or more LA structures are observed. In this case, counting may cease after completion of the grid opening that contains the 50^{th} LA structure.
- A total of 100 grid openings are counted without reaching the target sensitivity or observing 50 LA structures. In this event, the analysis should stop after completion of the 100^{th} grid opening.

TEM Data Deliverable

All data on the number, type and size of LA fibers observed during TEM analysis in the laboratory will be provided as an electronic data deliverable (EDD) using the most recent version of the spreadsheet developed for this purpose ("TEM Duff.xls"). The results for each sample will be expressed in terms of LA fibers per gram duff (dry weight), and also in terms of grams of LA per gram of duff (dry weight).

6.3 PLM Analysis

If analysis by PLM is called for in the project-specific SAP, the analysis will be performed on an aliquot of the ashed and homogenized residue using method PLM-VE as detailed in the most recent version of SOP SRC-LIBBY-03. PLM-VE is a semi-quantitative analytical method for asbestos that utilizes Libby-specific reference materials to allow assignment of samples into one of four "bins", as follows:

- Bin A (ND): non-detect
- Bin B1 (Trace): LA detected at levels lower than the 0.2% reference material
- Bin B2 (<1%): LA detected at levels lower than the 1% reference material but higher than the 0.2% reference material
- Bin C: LA detected at levels greater than or equal to 1%

A potential limitation to this approach is that the site-specific reference materials are based on LA in soil, not LA in ashed residue. This may introduce additional uncertainty into the results, but no reference materials based on ashed residue are presently available.

PLM-VE results will be recorded using the most recent version of the Libby site-specific EDD spreadsheet for PLM-VE analysis ("PLM (VE & PC) Data Sheet and EDD.xls").

7.0 QUALITY ASSURANCE

7.1 Field-Based Quality Assurance

Field Duplicates

Field duplicate duff samples will be collected at a frequency specified in the project-specific SAP. In the absence of such specification, the rate should be no less than 5%. Each field duplicate should be collected from a location close to the primary sample, and from an area of approximately equal size. Field duplicate samples should be labeled with a unique identifier. Sample details should be recorded on the appropriate soil FSDS, including the unique identifier of the "parent" field sample. Field duplicates are used to evaluate the sampling and analysis variability across duff samples. Unless indicated differently in the project-specific SAP, samples will not be qualified purely as a result of the difference between measured values between original and duplicate pairs.

7.2 Laboratory-Based Quality Assurance for TEM Analyses

Drying Blanks

For the purposes of this analysis, a drying blank will consist of one clean aluminum pan placed empty into the drying oven along with pans containing field samples of duff. After drying the duff samples, the clean tray will be removed and the surface will be rinsed with about 100 mL of FDI water into a clean container, which in turn will be filtered and prepared for TEM analysis. Detection of fibers on the drying blank filter will be taken as an indication of potential cross-contamination during drying.

Drying blanks should be prepared at a rate specified in the project-specific SAP. In the absence of a project-specific specification, drying blanks should be prepared at a rate of one per day that drying of samples is occurring. Unless indicated differently in the project-specific SAP, if the drying blank reports LA fibers, all samples in that drying batch will be assigned a qualifier to indicate the potential for cross-contamination.

Laboratory Blanks

A laboratory blank is a filter that is prepared by processing a clean crucible in the same way that a duff sample is prepared. That is, a clean crucible is treated by addition of FDI water and HCl, as described above. The contents of the crucible are then rinsed out, diluted to 100 mL, and an aliquot at least as large as the highest volume aliquot for the sample set is removed and used to prepare a filter for TEM examination. This type of blank is intended to indicate if contamination is occurring at any stage of the sample preparation procedure.

Laboratory blanks should be prepared at a rate specified in the project-specific SAP. In the absence of a project-specific specification, laboratory blanks should be prepared at a rate of 3%. Unless indicated differently in the project-specific SAP, if the laboratory blank reports LA fibers, all samples in that analytical batch will require re-preparation.

Filtration Blanks

A filtration blank is a clean filter that is prepared by passing 100 mL of laboratory FDI water through it. The purpose of this type of blank is to ensure that the filters are not contaminated in the laboratory, and that fluids used for diluting and processing samples are fiber-free.

Filtration blanks should be prepared at a rate specified in the project-specific SAP. In the absence of a project-specific specification, filtration blanks should be prepared at a rate of 2%. Unless indicated differently in the project-specific SAP, if the laboratory blank reports LA fibers, all samples in that analytical batch will require re-preparation.

Laboratory Duplicates

Laboratory duplicates will be prepared by applying a second aliquot of ashed residue suspension to a new filter, which is then prepared and analyzed in the same fashion as the original filter. The frequency of laboratory duplicates should be specified in the project-specific SAP. In the absence of such specification, the rate should be no less than 5%. Unless indicated differently in the project-specific SAP, samples will not be qualified purely as a result of the difference between measured values between original and duplicate pairs.

Recounts

The precision of TEM sample results should be evaluated by recounting selected grid openings in accord with the requirements specified in the most recent version of LB-000029.

7.3 Laboratory-Based Quality Assurance for PLM-VE Analyses

Laboratory Duplicates

Laboratory duplicate PLM-VE analyses will be prepared by examining a second aliquot of ashed and homogenized residue. The frequency of laboratory duplicates should be specified in the project-specific SAP. In the absence of such specification, the rate should be no less than 5%.

Unless indicated differently in the project-specific SAP, samples will not be qualified purely as a result of the difference between measured values between original and duplicate pairs.

8.0 REFERENCES

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ATTACHMENT A

FIELD SAMPLING DATA SHEET (FSDS)

Sheet No.: Duff- _____

LIBBY FIELD SAMPLE DATA SHEET (FSDS) rev0
DUFF

Field Logbook No: _____ Page No: _____
Station ID: _____ Sampling Date: _____
GPS Coordinate System: _____ Elevation Coordinate System: _____
X coordinate:_____ Y coordinate:_____ Elevation:_____
Sampling Team: _____ Sampler Initials: _____
Station Comments: _____

Data Item	Sample 1	Sample 2	Sample 3
Index ID (place pre-printed label in field provided)			
Sample Time (hh:mm)			
Sample Type (circle one):	Grab Composite # of Composites: _____	Grab Composite # of Composites: _____	Grab Composite # of Composites: _____
Field QC Type (circle one):	FS (field sample) FD (field duplicate) For FD, Parent ID: _____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____
Field Comments:			
Entered by (Provide initials):		**Validated by (Provide initials):**	

For Data Entry Completion (Provide Initials)	Completed by	QC by

ATTACHMENT B

DUFF PREPARATION SAMPLE DATA SHEET (PSDS)

LIBBY DUFF PREPARATION SAMPLE DATA SHEET (PSDS)

Laboratory Name: _____ Lab Job No.: _____ Lab QC Batch No.: _____ SOP: DUFF-LIBBY-OU3 (Rev 0)

Preparation by: _____ Preparation Date: _____

Drying Oven Temp. (°C): _____ Muffle Furnace Temp. (°C): _____ HCL Reagent Tracking No: _____

	SAMPLE INFORMATION			DRYING						ASHING				FILTER PREP			
	Index ID	Lab Sample ID	Mass (g), as received	Tray ID(s) used in drying	Tray weight (g)	Mass (g), during drying [tray + sample]			Mass (g), after drying [sample only]	Pan ID(s) used in ashing	Pan weight (g)	Mass (g), after ashing [pan + sample]	Mass (g), after ashing [sample only]	Mass of ash (g) taken for analysis	Volume of HCl added (mL)	Aliquot volume (mL)	Notes
						Check 1	Check 2	Check 3									
Example	X-12345	026589	500.3	A	5.71	63.12	55.90	55.84	50.13	A	15.87	36.98	21.11	0.26	15.7	1.0	
	↓	↓	↓	B	4.99	70.56	63.02	63.11	58.12	B	16.20	44.05	27.85				
	↓	↓	↓	C	5.23	89.63	71.85	72.03	66.8								

Note: All mass measurements should be recorded to an accuracy of ± 0.01 g.

QA Check by: Date: